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VOLUME 22, NUMBER 6

December 1961

American Industrial Hygiene Conference
Park-Sheraton Hotel, Washington, D.C.
May 13-17, 1962



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Industrial Benzene Exposure from Petroleum Naphtha: I. Rubber Coating Industry

LEONARD D. PAGNOTTO, A.B., HERVEY B. ELKINS, Ph.D.,
HEINRICH G. BRUGSCH, M.D., and JANET
E. WALKLEY, M.S.

*Division of Occupational Hygiene, Massachusetts Department of Labor
and Industries, Boston, Massachusetts*

A study was made of the benzene exposure of workers in the rubber coating industry where large quantities of certain naphtha solvents containing varying amounts of benzene are used. The benzene exposure of churnmen, spreaders and saturators as measured by air tests, and urinary phenol determinations ranged from low grade to excessive. In cases where the MAC was exceeded the benzene content of the solvent was more than 3%.

Introduction

BENZENE is widely recognized as one of the most harmful of the common solvents. Its deleterious and sometimes fatal effects, manifested mainly in the blood forming tissues of the body, are well documented. The subject of a safe exposure level to benzene has also been extensively studied and continues to receive considerable attention. The findings in recent years have resulted in several downward revisions in standards of permissible concentrations. The value presently used in Massachusetts and one generally accepted is 25 parts of benzene per million parts of air (by volume) (ppm).

The incidence of benzene intoxication in this country has been greatly reduced by the substitution of less toxic solvents in industries such as artificial leather manufacture, rubber products manufacture, and printing. In our experience benzene as such is now rarely used in large quantities as an industrial solvent.

Benzene is known, however, to be present in varying amounts in certain widely used and moderately volatile petroleum naphthas.¹ Yet, little attention has been paid to the possible benzene hazard associated with the use of such naphthas. It has apparently been commonly assumed that if the naphtha exposure is controlled, that the benzene will automatically be taken care of. Based on the current MAC's (Maximum Allowable concentration) for benzene and naph-

tha of 25 and 500 ppm, respectively, the benzene hazard would in theory exceed that of the naphtha only when the latter contained more than 5% benzene. There is, however, some question about the soundness of this assumption. Hammond and Hermann concluded from their study that naphtha containing in excess of 2.5% should be handled with additional precautions.² Because of the more serious consequences of benzene intoxication, in comparison with the usually transient effects of naphtha, consideration of the benzene exposure resulting from the use of naphtha with benzene contents below 5% would be of value.

Rubber Coating Industry

The rubber coating industry is a heavy user of petroleum naphtha and is ideal for such a benzene exposure study. Several hundred workers in our Commonwealth receive a daily continuous exposure to benzene from petroleum naphtha solvents during the process of manufacturing rubber coated products.

From the solvent exposure point of view this process begins in the churn room. Here in some Massachusetts plants, 1000 gallons or more of naphtha are pumped into mixing churns each day. The charge in a single churn varies, but a formulation frequently used may consist of 700 to 800 lbs of rubber stock and other solid ingredients and in addition, 80 to 110 gallons of naphtha solvent. After six to eight, or as long as 24 hours of mixing, the prepared stock is drawn into vats or barrels. Additional solvent is usually added and the mix is mechanically stirred to obtain a desired consistency before be-

This paper was presented at the Twenty-second Annual Meeting of the American Industrial Hygiene Association, Detroit, Michigan, April 12, 1961. The work reported was supported by the U. S. Department of Health, Education and Welfare through Public Health Service Research Grant 5503.

ing delivered to the saturator or spreader machines.

Some material must be pretreated before receiving its final rubber coat. This is done in the saturating room. On the saturating machine, (Figure 1), fabric is unrolled through a light rubber mix in a trough supplied from a feeder vat or barrel, and then passes on to a dryer and is rerolled.

The final rubber coat is applied by the spreading or coating machine (Figure 2). Here a fairly heavy rubber mix is added to the fabric with a ladle or by gravity feed. Frequently, the feed stock has a bread dough consistency and the worker is able to handle the mix with cupped hands. A doctor blade spreads and regulates the thickness of the coating and the fabric then passes over a dryer and is rerolled.

Procedure and Methods

Our industrial hygiene study covered eleven rubber coating plants. This included practically



FIGURE 1. Saturator machine.

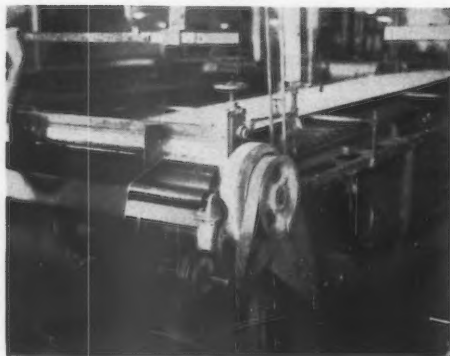


FIGURE 2. Spreader machine.

all the large Massachusetts plants in this industry and some of the smaller ones.

Petroleum solvents with boiling ranges that encompass the boiling point of benzene were collected without any reference to their original suppliers in order to obtain an overall picture of the benzene content of naphthas in current use. The solvents were analyzed by an ultraviolet absorption procedure. Isopropyl alcohol was used as the diluent except in cases where the presence of large amounts of toluene and other high boiling constituents interfered with the analysis. For these solvents an aliquot was added to a 1:1 solution of cyclohexane (boiling point 81.5°C) and heptane (boiling point 98.5°C) and passed through a 30-inch fractionating column. The benzene was recovered (95% or more) in the cyclohexane distillate. Most of the toluene remains in the heptane residue.³

Air tests for benzene were made at the feed end of the saturator and spreader machines. The churn room air tests were of general exposure. Air samples were collected on silica gel in a U-tube at the rate of two liters per minute for 15 to 30 minutes. Because of the fire hazard a hand-operated midget impinger pump was used as a source of vacuum. The silica gel was ordinarily extracted with isopropyl alcohol and an aliquot was analyzed by an ultraviolet light absorption method. For samples containing large amounts of toluene the silica gel was transferred to a distilling flask, a 1:1 solution of cyclohexane and heptane added, and fractionated as described for the solvents.³ Except when toluene vapor was present in large excess, a satisfactory separation was achieved.

Urine samples were collected from workers and analyzed for total phenol. Teisinger and Fiserova-Bergerova⁴ studied the phenol in urine of benzene workers. We confirmed their findings in our laboratory and found the phenol test to be a reliable index of benzene exposure with a sensitivity greatly exceeding that of the urine sulfate ratio test.

The method of analysis was that proposed by Theis and Benedict⁵ but modified by our laboratory.⁶ Urine samples were treated by acid digestion, steam distilled, and analyzed with diazotized p-nitro-aniline.

For a worker exposed to benzene the phenol excretion increases to a maximum after a few hours of work, but returns to normal or nearly so before he returns to work the following day. Because of this diurnal variation it was essential to collect the urine samples toward the end of the work shift. Urinary phenol concentrations adjusted to a specific gravity of 1.024 in excess of 200 mg per liter were taken as indicating an excessive exposure to benzene.⁷

Blood examinations consisting of white counts, differentials, (including an estimation of the number of platelets present) and hemoglobins, were done on workers in plants where benzene exposures were found to approach or exceed the MAC.

Results and Comments

Table I summarizes our results on the benzene analyses of 32 naphtha solvents. The benzene ranged from 1.5 to 9.3% by weight. About one third of them come under the Massachusetts Labeling Regulations, which require a benzene warning label when the benzene content exceeds 3%.

Atmospheric benzene and urinary phenol excretion results for spreaders, saturators, and churn men are listed on Tables II and III.

Excessive benzene exposures in our survey were found at saturators in three of only four plants doing this operation every day. On one occasion the concentration of benzene vapor was as high as 125 ppm. The urinary phenol excretion of the saturator workers in these three plants were also the highest we reported, ranging from 370 to 917 mg per liter of urine.

Although in each of these plants the saturator machines had enclosed and mechanically exhausted dryers, the operator's exposure to naphtha solvent vapors from the highly diluted mix generally used in this process was found with the combustible gas detector to be as high as 1500 ppm. The benzene contents of the naphthas in use were 6.2 and 9.3% by weight.

Additional ventilation was recommended for adequate control of the benzene and naphtha exposure. In one plant local exhaust ventilation was installed at the feed end of the saturator and a change to a naphtha containing less benzene (3%) was made. On a follow-up visit the benzene exposure was found to have been reduced to about 7 ppm and urinary phenol, to less than 70 mg per liter.

The benzene vapor concentrations in churn rooms were somewhat lower than one would expect from the urinary phenol values found on the churn men. This can be explained by the fact that it was not always possible to be on hand to make air tests of such activities as loading and unloading churns, cleaning vats, and other churnroom activities that are done intermittently. It was apparent, however, from readings obtained with the combustible gas apparatus that the high-speed mixer was an important source of exposure. Readings of 900, 500 and 350 at the top of the tub level, breathing level of the operator, and floor level respectively were reported in one plant and these are typi-

cal findings. Urinary phenols in excess of 200 mg per liter were found for one or more churn men in five plants.

Ventilation in churn rooms was usually of the

TABLE I
Benzene Content of Naphtha Solvents

Number Solvents Analyzed	Benzene % By Wt.
15	1.5-2.5
6	2.6-3.5
11	3.6-9.3

TABLE II
Number of Tests for Phenol in Urine

Churn Men	Spreaders	Saturators	Urinary phenol mg./liter*
6	8		0-50
15	22	9	51-100
6	25	2	101-150
8	23		151-200
3	9		201-250
4	7		251-300
1	3		301-350
2		3	351-400
1		1	401-500
		3	501-700
		1	917

* Adjusted to a Specific Gravity of 1.024

TABLE III
Number of Tests for Benzene in Air

Churn Men	Spreaders	Saturators	Benzene ppm
3	9	2	0-5
1	11	1	6-10
5	9	2	11-15
	6		16-20
	5	1	21-25
	1		26-30
	1	1	31-35
		2	36-40
		3	50-70
		1	90
		1	125

TABLE IV
Use of Combustible Vapor Detector to Calculate Benzene Concentrations in Air

Vapor Detector Reading	Benzene in Solvent % by Wt.	Benzene in Air ppm	
		Calculated	Actual
1400	5.6	77	125
500	6.2	31	60
280	1.5	4	9
200	5.6	11	16
85	3.1	3	5

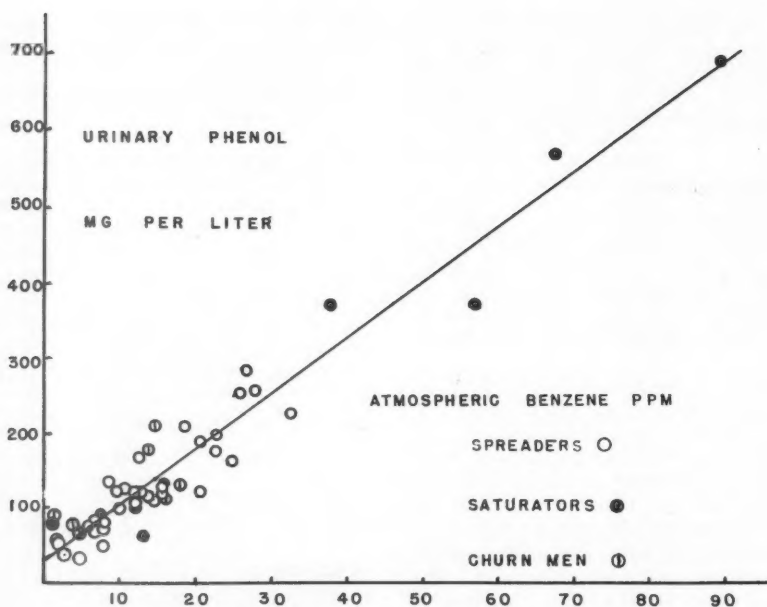


FIGURE 3. Comparison of phenol in urine with benzene in air.

TABLE V
Blood Examination Results

	Plant A	Plant B	Plant C
	Number of Tests		
	32	9	6
1. Hemoglobin (gm)			
Over 13.5	27	8	6
Under 13.5	5	1	0
2. Erythrocytes (millions/cmm)			
Over 4	30	8	
Under 4	2	1	
3. Leucocytes (per cmm)			
Over 10,000	1	0	2
5-10,000	31	9	4
4. Lymphocytes (per cent)			
Over 50	0	0	0
Under 50	32	9	6

The number of platelets in each case was considered to be within normal limits.

general type and consisted of window and ceiling fans. In some plants, however, local exhaust ventilation was provided to churns but in practically no case was any special ventilation provision made for the high-speed mixing areas.

The extent of benzene exposure of spreader operators was slightly lower than that of churn

men. In only four plants where air tests were made did we find excessive exposures. The highest benzene air value was 32 ppm. The urinary phenol values were consistent with these findings.

Ventilation on spreaders was usually of the canopy-type located over the dryers but in some plants no local exhaust ventilation was used. Plants in this latter group using naphtha with a benzene content in excess of 3% accounted for most of the high benzene exposures reported for spreaders.

Readings with the combustible gas detector were made in several plants. The concentration of benzene vapor was calculated on the basis of the benzene content of the solvent. Table IV shows that the value arrived at on this basis indicated only about half the actual benzene present.

Figure 3 compares benzene air values with urine phenol findings on saturators, spreaders and churn men. Each point indicates the average phenol excretion of workers in a work area and the average benzene concentration found on the same day. The urine specimens were spot samples collected toward the latter part of the work shift. It is apparent from the air-urine correlation that the phenol test is a good index of benzene exposure.

Medical Findings

The results of blood examinations of 47 men including spreaders, saturators, and churn men in three plants are summarized in Table V. In plant A, five employees show a lowered hemoglobin (below 13.5 grams). One man in this group had a 11.3 gram hemoglobin and 3,950,000 red blood count. This worker, a 28 year old man, has been employed for about three years in the churn room preparing mixes for the saturating machine. Next to the saturator operator he had also the highest phenol excretion in the plant, a value of 480 mg per liter of urine.

In plant B the blood findings were normal except for one worker. He was a 58 year old man who was seen by his physician because of a general malaise and weakness. It was found that he suffered from a profound anemia (hemoglobin 7.5 grams). Hospital observation showed an erythroblastic bone marrow, and the possibility of leukemia was raised. However, after being taken off his job and given iron therapy, he recovered fully. This man was employed for 18 years on an insole callender machine, which rolls rubber stock to a desired thickness then passes it into a trough where cement is applied to one side. Eighteen months before his illness cement made with naphtha was introduced. Since several major changes had been made in the process before it was suspected that his solvent exposure on the job might be the cause of his illness, we were unable to determine the actual benzene exposure in this case.

No abnormal findings were reported in plant C.

Summary

Benzene is present in certain naphtha solvents in amounts varying up to 9.3% by weight. The extent of benzene exposure from these naphthas in the rubber coating industry was determined by air tests and urinary phenol analyses. This latter test is a sensitive and reliable index of benzene exposure. It has a sensitivity greatly

exceeding that of the urine sulphate ratio test and enables detection of benzene exposures well below the present MAC for this substance.

Excessive benzene exposures (one case as high as 125 ppm) were consistently found on saturators using naphthas containing considerably more than 3% benzene.

The benzene exposure of churn men was much lower than that of the saturators. Phenol excretion results were mainly below 200 mg, however, the range of values extended to 480 mg per liter of urine.

The extent of benzene exposure for spreaders was slightly lower than that of churn men. Air test results ranged up to and slightly above the MAC. A number of urine phenol values were above 200 mg but none above 350 mg per liter.

The blood examinations thus far have revealed that several workers showed hemoglobins below 13.5 grams and one man suffered a marked blood dyscrasia that subsided completely following withdrawal from work. A complete report of our blood findings will follow at a later date.

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RESEARCH IN AIR POLLUTION

THE FOURTH EDITION of *Guide to Research in Air Pollution* has been published by the American Society of Mechanical Engineers. The volume was prepared by a Task Group created by the ASME Committee on Air Pollution Control's Subcommittee on Current Projects and Trends. This fourth Edition of the Guide covers projects active in the calendar year 1959. It may be obtained by writing ASME Order Department, 345 E. 47th Street, New York 17, New York. Price of the Guide is \$4.50.

A New Technique for Evaluating Respirator Performance

WILLIAM A. BURGESS, S.M., LESLIE SILVERMAN, S.D.,
and FELIX STEIN, S.B.

Harvard School of Public Health, Boston, Massachusetts

Evaluation of respirators used for protection against dusts, fumes, and mists significantly more toxic than lead can be conducted utilizing a uranine aerosol with a geometric mean size of 0.20 micron. The uranine aerosol is introduced into an exposure helmet chamber which permits exposure of the subject wearing the respirator to concentrations of below 4 mg/m³. The aerosol is collected continuously outside the respirator and during inhalation in the respirator void with membrane filters. Uranine analysis by fluorescence permits evaluation of respirator penetration as low as 0.05% in a fifteen minute study with an exposure concentration of 3 mg/m³.

Introduction

RESPIRATORY protective equipment (respirators) possess demonstrated value as emergency control devices and as supplements to conventional engineering control of toxic aerosols.^{1, 2} There is, however, a need for evaluating the protection provided by respirators under field conditions. A limited study of the actual leakage during use on subjects of half and full face masks by one of the authors³ (during World War II) indicated that leakage values on half masks may range from one to 20 per cent and from less than one to five per cent on full face masks. This degree of protection is not adequate for many of the applications to which respirators are now being applied.

Present U. S. Bureau of Mines specifications for the design and testing of respirators are based on dusts, fumes, and mists not significantly more toxic than lead.⁴ We are now exposing individuals to much more toxic materials and thus the performance of available respiratory devices should be critically reviewed to assess their adequacy for such application extension beyond the present Bureau of Mines schedule.⁵ The toxicity levels with which we are concerned in handling materials or working near nuclear processes are often several orders of magnitude more restrictive than the level for lead. To illustrate, non-radioactive beryllium is considered to be 100 times more toxic than lead whereas the radioactive nuclides plutonium-239, polonium-210, and strontium-90 are respectively

considered five million, two billion and three billion times more toxic on a weight basis. Under certain operational and emergency conditions respiratory protective equipment is now used for reducing the inhalation of such toxic aerosols. It is therefore imperative that there should be developed an evaluation procedure for rating and qualifying respirators on wearers under conditions comparable to exposure to highly toxic radioactive materials such as plutonium and non-radioactive materials such as beryllium. The quantitative technique proposed in this paper satisfies these requirements.

In approaching the design of such an evaluation procedure guidance was available from the tentative specifications set forth by the Subcommittee on Requirements, AEC Committee on Respiratory Protective Equipment in their report of April 14, 1959.⁶ The primary specification of the AEC Committee was that the overall penetration should not be greater than 0.10 per cent for a full face mask and not greater than 1.0 per cent for half masks. This penetration was based on testing with a standardized homogeneous particulate aerosol in the size range of 0.1 to 0.5 micron diameter. It was defined as total penetration from all sources such as filter, valves, speaking diaphragm and face seal.

Leakage has been determined in the past largely by chamber tests, although at present the U. S. Bureau of Mines is experimenting with a dioctyl phthalate (DOP) peripheral seal test. The British, at their Chemical Defense Center at Porton and our Chemical Corps Bacteriological Warfare Laboratory at Fort Detrick, have developed a mask leakage evaluation tech-

This work done under Contract No. AT(30-1)2355 between the Atomic Energy Commission and Harvard University. Opinions expressed are those of the authors and not necessarily those of the Atomic Energy Commission.

nique using BG spores of approximately one micron diameter as a test aerosol and a cotton collector inserted in the mouth or an inner mask filter attached by adhesive tape to the face to determine penetration.⁷

Test methods available to date suffer from many obvious deficiencies when considered for evaluation of respirators worn for protection against highly toxic aerosols. The sizes of previously used suspensions are generally greater than one micron and this does not necessarily predict the leakage of smaller sizes. Test procedures commonly used require the addition of hoses or appurtenances to the mask body which modify the designed weight and suspension of the respirator. Subjective reactions to leakage, a common method of evaluation, is acknowledged to be qualitative due to differences in the sensory response of subjects.

An ideal respirator leakage test method requires lightweight and non-obstructive equipment capable of measuring aerosol leakage in a representative manner. This method should appraise leakage during inhalation periods only. A basic requirement is to detect less than 0.10 per cent penetration with an aerosol having a geometric mean size of 0.1 to 0.5 micron. In addition to predicting percentage penetration or leakage, the method should also permit determination of absolute penetration in weight of aerosol. By definition this requires determining the respiratory minute volume of the wearer. Since the procedure is intended for use by AEC establishments, the method should be developed from equipment available at such facilities. For field use the equipment should be lightweight and portable.

Evaluation Technique

In the design of the equipment it was necessary to consider the following factors, each of which will be discussed in detail: the aerosol, aerosol generation, the exposure chamber, the aerosol sampling system, and the measurement of respiratory air flow volumes.

The Aerosol

The general characteristics desired in the aerosol include: (a) ability to generate an aerosol in the size range of 0.1 to 0.5 micron diameter at adequate air concentrations, (b) detection sensitivity which will permit defining penetration of less than 0.10 per cent in a 15 minute test, (c) non-toxic in the concentrations and for the periods used, and (d) relatively low cost.

Initially, attention was given fluorescent pig-

ments and dyes since they appeared to possess these minimum desirable characteristics. A number of pigments were attractive but they required moderately toxic and volatile solvents for solution and dispersal. These solvents presented manifold difficulties since they were not compatible with materials used in certain aerosol generators under study, their use resulted in exposure of the test subject to a moderately toxic vapor, and they presented a fire and explosion hazard.

Uranine, a commercial dyestuff previously used as a tracer in air pollution work,⁸ possessed the same desirable characteristics without the liabilities inherent in fluorescent pigments. This disodium salt of fluorescein is readily soluble in water insuring compatibility with all generation and sampling equipment. It can be efficiently collected on membrane filter media and is easily leached for analysis. Uranine is excited by blue light of 4400 to 5100 Å and emits at 5100 to 5900 Å. Utilizing a Photovolt Multiplier Fluorescence Meter (Model 520 M) and a Photovolt Fluorescence Unit (Model 54) with proper selection of light source and filters, concentrations as low as 0.1 nanogram of uranine per ml of water can be determined.

The following instrumental configuration gave us the highest sensitivity with the lowest background. The exciting light source is a 100W high pressure mercury vapor lamp with predominant lines at 3650 Å, 4050 Å and 4360 Å. This light is filtered by a B Hg 1-2-3 filter which transmits all three wave lengths plus approximately 500 Å beyond the third band. Therefore, the light reaching the uranine sample has a maximum wave length of 4860 Å covering the upper end of the band for the excitation of the uranine (4400 Å to 5100 Å). The light emitted from the uranine sample has a wave length of 5100 Å to 5900 Å. This light is filtered by a B 540 filter which passes light from 4900 Å to 5900 Å. This filter arrangement allows all light emitted from the uranine to reach the photomultiplier tube but cuts out stray light from the exciting light beam.

In addition, an opal glass is inserted in front of the photomultiplier tube to distribute the light evenly and to reduce the effect of reflection from the glass cuvettes.

A linear relationship (on log-log plot) is obtainable from 10^{-8} gm/ml to 2×10^{-6} gm/ml. The calibration curve, shown in Figure 1, is reproducible within five per cent.

Aerosol Generation

For the generation of particles with a geometric mean size (on a count basis) in the range

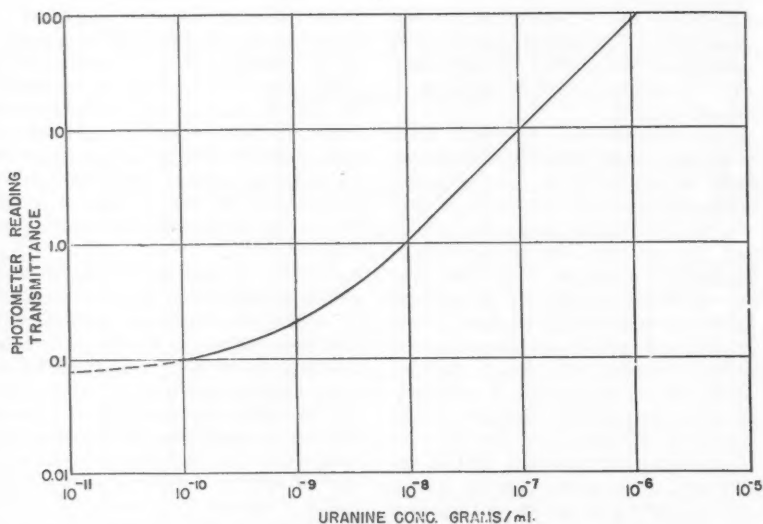


FIGURE 1. Fluorescence of uranine solutions versus uranine concentrations.

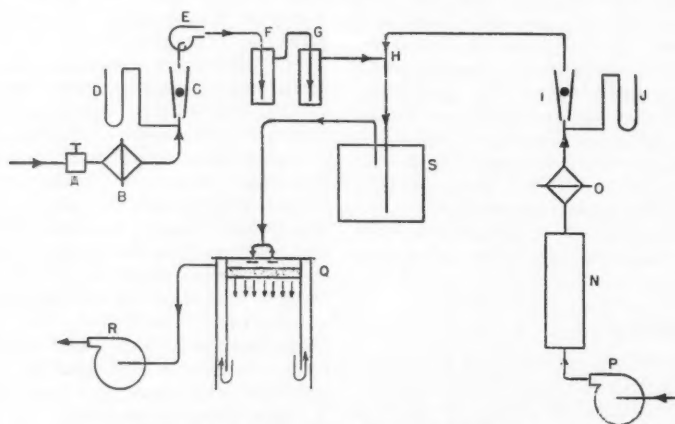


FIGURE 2. Uranine aerosol generation system.

of 0.1 to 0.5 micron, several commercially available nebulizers were tested using uranine as the aerosol material.

The aerosol generation technique finally adopted was based on an all glass Pen-i-Sol nebulizer (Hudson Oxygen Therapy Sales Co., Los Angeles 27, Calif.). The generation technique is shown in Figure 2. A 2.35 per cent solution of uranine is placed in the Pen-i-Sol nebulizer (E) which is driven by pre-filtered compressed air. The air flow rate through the generator is approximately 3.5 liters per minute

(lpm). The aerosol generated by the nebulizer is then passed through two standard Greenberg-Smith impingers (F and G) which remove the large particles from the air stream. Dilution air which has been dried (N) and filtered (O) is then metered (I) and mixed with the aerosol in the initial mixing chamber (H). After additional mixing of the generated aerosol and dilution air in a second drying chamber, the diluted test aerosol is directed to the exposure chamber.

Particle size analysis of the aerosol supplied to the exposure location was conducted by the col-

lection of the aerosol on carbon coated electron microscope screens. The collected aerosol was viewed under the electron microscope and sizing was obtained from electron micrographs. A geometric mean particle size of 0.2 micron with a standard deviation of approximately 2.0 was consistently generated with the above procedure.

Exposure Chamber Helmet

In order to expose a man wearing a respirator to the test aerosol while in a work situation an exposure chamber with certain unique design features was required. The exposure chamber, in order to perform its basic function, must provide a known concentration of aerosol to the breathing zone of the subject. In addition, the chamber must permit exposure of the subject without surrendering his mobility to perform various work activities during the test program. The chamber must also be designed to permit various connections to the respirator, provide full vision for the subject, and allow the removal of the test aerosol. This latter feature seemed especially desirable with the sensitive analytical procedure in use since trace contamination of laboratory equipment could result in erroneous test results.

An exposure helmet incorporating the basic design features listed above was designed and is shown in Figure 3. It consists of a rigid platform (A) designed to allow easy assembly of two wrap around concentric and transparent walls (B). The aerosol is introduced through two ports (C) and distributed by impaction plates (D) in a shallow plenum (E). The aerosol then passes through an open cell polyurethane foam pad (F) (Scott Paper Co., Foam Division, Grade S-101-15-80, 80 pores per inch) which insures a homogeneous distribution of aerosol in the chamber. From the exposure zone the aerosol is exhausted out of doors through the annulus formed by the two plastic cylinders. Connectors for tubing leads to the respirator are provided at (G).

The chamber design ensures homogeneous distribution of the aerosol in the exposure zone of the test subject. Tests have been conducted which demonstrate a flat concentration profile with maximum variation in concentration of less than 10 per cent in the exposure zone. The concentration of uranine in the helmet can be adjusted to a maximum of 4 milligrams of uranine per cubic meter of air.

The helmet can be positioned in a fixed location with an instrument stand and boom or it can be attached directly to the subject with a suitable helmet frame.

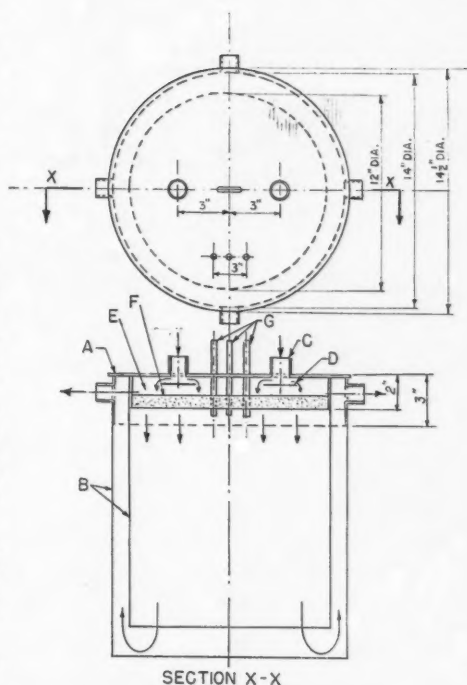


FIGURE 3. Exposure chamber helmet.

Aerosol Sampling System

Membrane filters, chosen for the collection of uranine, were found to be greater than 99.5% efficient on this size aerosol. Filter holders, required for sampling in both the exposure helmet and the respirator, must permit representative air sampling in the respirator void or dead space without interfering with normal use of the respirator by the subject. For this purpose the holders must be small and light weight. The final filter holder, shown in Figure 4, is about the size of a quarter and weighs only 6 gms. It has an aluminum body, porous "Kel-F" backing plate, and integral hypodermic needle suction take-off. A screw top insures a tight filter seal so that "by-passing" of the membrane filter does not occur. In preparing for a test, the hypodermic needle is pushed from the inside of the respirator through the rubber frame. The chamber filter holder is clipped to the inside of the chamber. Catheter tubing is used to attach the holder to the suction source.

Evaluation of performance of a respirator worn by a test subject in a test aerosol atmosphere can be determined by simultaneous air

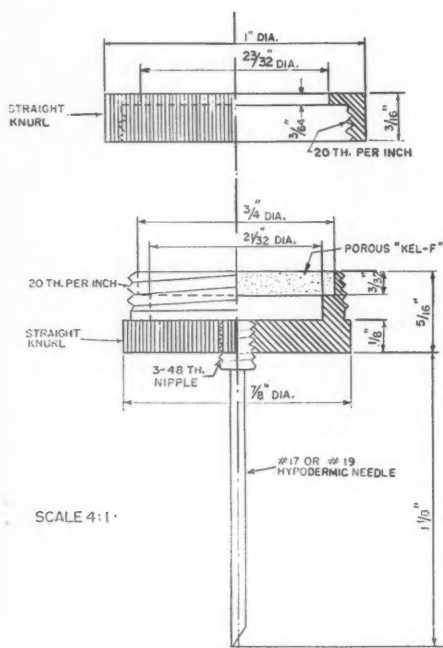


FIGURE 4. Filter holder.

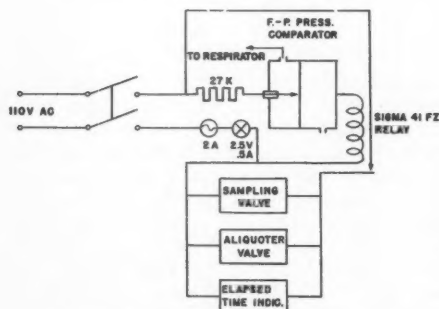


FIGURE 5. Respirator sampling director.

sampling in the exposure chamber and air sampling in the respirator dead space during the inhalation phase of the test subject's respiratory cycle. Comparison of concentration of the aerosol outside to the concentration of the contaminant inside the respirator can be expressed as percentage of contaminant penetration.

A respirator sampling director developed for this study permits inside respirator sampling during either inhalation or exhalation. The operating principle of the unit can best be de-

scribed by reference to Figure 5. A sensitive pressure switch, shown as F-P Pressure Comparator, is connected to the respirator dead space by light, noninterfering, small bore plastic tubing connected to a hypodermic needle which penetrates the respirator body. During inhalation the diaphragm of the pressure switch closes the control circuit which opens a sampling valve and permits an air sample to be taken from within the respirator. At the start of exhalation the circuit is opened, the sampling valve closes, and respirator sampling ceases. The respirator sampling time is determined by an elapsed time indicator which is also activated during the inhalation period. The total air volume is determined from the total sampling time and flow rate through a calibrated critical orifice in the sampling line.

The sampler determining the aerosol concentration outside the respirator samples continuously. The total air volume sampled is determined from the total time of the test and flow rate through another critical orifice sampling line. The two sampling systems showing the respirator sampling valve, critical flow orifice, and common suction source are shown in Figure 6.

The air volumes sampled and the amount of uranine on the sampler filters permit computation of the air concentration of the uranine aerosol inside and outside the respirator. The percentage penetration is determined from this information.

The respirator sampling director also provides a means for measuring the respiratory minute volume of the subject. The product of the respiratory minute volume and the aerosol concentration inside the respirator defines absolute penetration or the quantity of aerosol which has penetrated the respirator and has been inhaled by the subject.

Determination of Respiratory Air Flow Volumes

The respiratory minute volume is determined by means of a parallel air path of high resistance to the main path through the respirator filter or cartridge. The flow in the aliquoting path is in fixed ratio to the total respiratory flow for a given respirator and filter design. The parallel air flow path, shown in Figure 7, consists of a hypodermic needle inserted in the mask, a capillary resistance, a solenoid operated valve directed by the pressure sensing device which opens only during inhalation, and a soap film spirometer which acts as a flow integrating device. During the inhalation phase the valve opens and an aliquot of air passes through the capillary into the respirator. The soap film

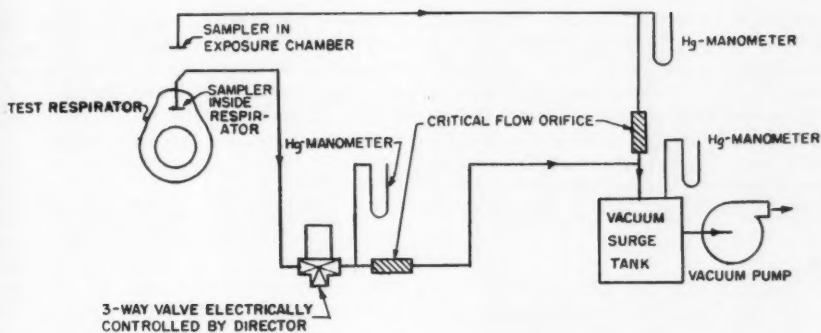


FIGURE 6. Aerosol sampling system.

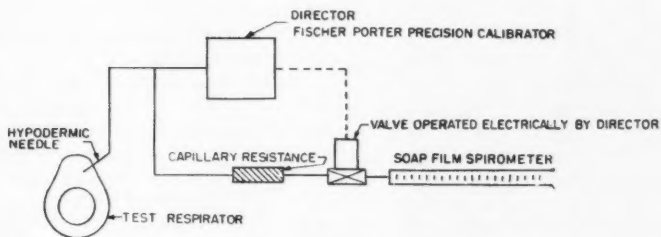


FIGURE 7. Respiratory air flow volume indicator.

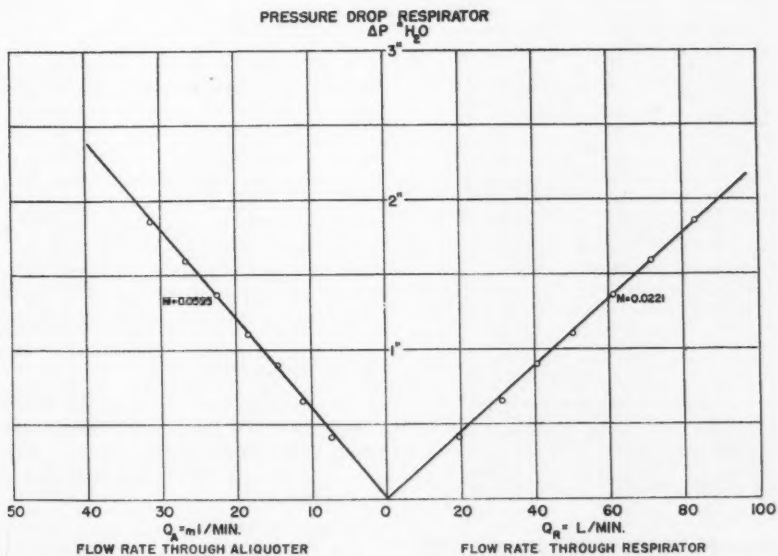


FIGURE 8. Aliquoter calibration for a standard respirator.

spirometer defines this air volume. Since this air path is closed during exhalation the soap film moves stepwise inward progressing only during inhalation. A calibration for each respirator is made by sealing the respirator to a test manikin with the aliquoter in place. Flow through the respirator and aliquoter are noted for varying inspiratory suction, under steady state flow conditions. A calibration plot is then made as

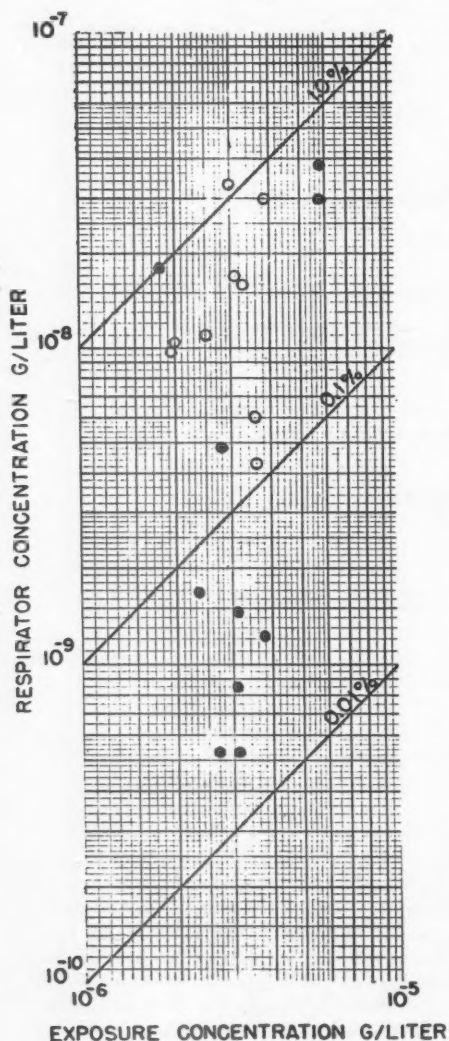


FIGURE 9. Respirator performance.

shown in Figure 8. Measurement of the minute volume for a subject under test is obtained by measuring the aliquoting volume with the soap film spirometer. This value, noted as Q_A on Figure 8, then permits direct assignment of respiratory flow from aliquoter calibration.

Application

The respirator evaluation technique described in this paper has been used to evaluate percentage penetration on both half mask and full mask filter respirators and on air-line respirators. The test schedule has included frame sealed tests on manikins with standard mechanical breathing pumps⁹ and subjects wearing the respirators at sedentary conditions and moderate work of 622 kilogram-meters per minute. The evaluation procedure has been shown to be applicable to all types of respiratory protective equipment.

Representative performance of a number of different models of half masks (open circles) and full face masks (solid circles) under work conditions of 622 kilogram-meters per minute is shown on Figure 9. No attempt was made, nor was it the concern of this study to conduct a comparative rating of all existing equipment. This study was undertaken to determine if all types of equipment could be evaluated by the proposed procedure.

Conclusion

A quantitative method of evaluating over-all leakage of respirators designed for protection against highly toxic aerosols has been developed for use while being worn by sedentary and working subjects. The method allows definition of penetration or leakage well below 0.1 per cent using an aerosol with a geometric mean size of 0.2 micron. The same procedure can be used for smaller sizes and longer periods of use. It can also be applied to existing aerosols in the field. The procedure has been applied to all available respirator models with complete success.

Acknowledgment

The authors wish to thank M. Corn for his assistance in the uranine aerosol generation and sizing techniques.

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CERTIFICATION DEADLINE

THE AMERICAN BOARD OF INDUSTRIAL HYGIENE, at its second annual meeting on October 27, 1961, considered those applications for certification which so far have been received. The first certificates will be issued in the near future. Industrial hygienists who have had fifteen years of practice with recognized contributions to the field, following acceptable education, are eligible for certification without examination upon application to and approval of the Board. Applications for certification without examination must be received prior to July 1, 1962. If the entry into full-time practice of the profession, or of a specialized aspect, was prior to July 1, 1947, applicants should file at once without waiting for their fifteen year anniversary.

Those having eight years or more of qualifying industrial hygiene practice following a suitable baccalaureate degree are eligible for certification on examination. The first examination will be held in May 1963, at a time and place convenient to those attending the American Industrial Hygiene Conference.

An informative booklet and application forms may be requested of Dr. Henry F. Smyth, Jr., Secretary-Treasurer, American Board of Industrial Hygiene, 4400 Fifth Avenue, Pittsburgh 13, Pennsylvania.

INDUSTRIAL VENTILATION CONFERENCE

NORTH CAROLINA STATE COLLEGE will be the host to the fourth annual Industrial Ventilation Conference on March 19-22, 1962. This Conference will provide three and one-half days of workshops for the presentation of design information and practical solutions to air handling problems. This will be a meeting place for the exchange of ideas and problem solutions for those interested in good industrial ventilation. Instruction will be by lecture, demonstration, and class problems. The Conference will be conducted by the Mechanical Engineering Department of the School of Engineering and the College Extension Division of North Carolina State College.

For further information address Motte V. Griffith, Jr., College Extension Division, North Carolina State College of Agriculture and Engineering, P. O. Box 5125, Raleigh, North Carolina.

Determination of Lead in Urine by an Ion Exchange Method

WILLIAM H. HILL, FAY H. HENGSTENBERG,
and CLAUDE E. SHARPE

*Department of Public Health Practice, Environmental Health Section,
Graduate School of Public Health, University of Pittsburgh,
Pittsburgh 13, Pennsylvania*

An ion exchange method for the determination of lead in urine is described which involves 1) the automatic feeding of urine into an ion exchange column; 2) the concentration of lead from urine by a suitable resin; 3) the elution of lead from the resin by an acid, and 4) the subsequent determination of lead by an acceptable method. The results obtained by this method from the urines of lead-exposed persons compare favorably with results obtained by other methods. Recovery in lead-spiked urine is 90 to 100%.

SINCE one may detect incipient cases of lead poisoning¹ in exposed industrial workers by lead urinalysis, it is desirable to have a method that is simple, troublefree, accurate and that requires a minimum of operational personnel, room and equipment. The older methods of precipitation² and ashing³ are laborious and require considerable laboratory facilities. The extraction methods of Tompsett⁴ and McCord and Zemp⁵ are more rapid but require a great deal of manual work.

Ion-exchange offers an accurate method requiring little laboratory equipment and bench space, and most of the operations can be controlled by automation. Work on a large number of samples may be carried out simultaneously without close supervision by the analyst.

The ion-exchange method for analysis of lead in urine involves: (1) automatic feeding of urine into an ion-exchange column; (2) the concentration of lead from urine by a suitable resin; (3) the elution of lead from the resin by an acid; and (4) the subsequent determination of lead by an acceptable method.

Apparatus

Ion-exchange column: We use 14 mm O.D. \times 10 cm long columns with fritted glass discs (Microchemical Specialties Co., Berkeley 3, California) with a Buchner type funnel fused to the top. We have, also, used a thistle tube with

a glass wool plug at the bottom to hold the resin, and rubber tubing with a screw clamp to control the flow of fluid. The glass wool is first soaked with HCl to remove lead. The style A ion-exchange column with special drip tip and ground glass joint on funnel (Microchemical Specialties Co.) is perhaps the most convenient since the column cannot run dry.

Automatic Feeding System: We use a liter (or other size) separatory funnel with ground glass stopper, with its stem dripping into the funnel of the ion-exchange column. The stem of the separatory funnel should be short and of large bore. With the well-greased glass stopper inserted and the stop-cock wide open, the funnel will feed into the ion-exchange column at the same rate as liquid is withdrawn from the bottom of the latter. Other automatic feeding devices can also be used.

Infra red lamp: Radiator (Fisher).

Polarograph of suitable sensitivity for determination of small amounts of lead. We use the Sargent XXI and follow the technique described by Meites.⁶ This is not a necessary part of the equipment or method. We have also made lead determinations using the dithizone method as described by Sandell⁷ with the same results as with the polarograph.

Reagents

2N HCl: Analytical reagent grade HCl, with the normality adjusted with deionized water.

Amberlite: IRC-50 ion-exchange resin (Rohm and Haas).

The work reported in this paper was done under National Institutes of Health Grant RG-4924, Electrochromatography in Hygiene and Air Pollution.

AgNO₃ solution: 0.1N used but not critical.
Acetone: Mallinckrodt AR or equivalent.

Procedure

The ion-exchange columns are prepared by filling them to a height of 6 to 8 centimeters with the IRC-50 resin. The resin is then soaked and washed with 2N HCl until there is no lead in the HCl wash from the resin, as tested with dithizone reagent. This is usually accomplished by two 10-ml portions of acid dripped very slowly through the resin, at about the rate of a drop every five seconds. The resin is then washed with deionized water to remove the acid. If there is no precipitate visible when the wash is added to *AgNO₃* solution, the columns are ready for use. From this time on the columns are not again permitted to run dry, but are kept covered with HCl or deionized water. Between analyses, it has been found to be good practice to cover the resin with HCl. This is, of course, washed off before the next analysis. For the amount of lead normally found in urine, no other treatment is necessary between analyses.

In the bottle in which the urine sample is to be collected, a few drops of Permachem PC (believed to be a tributyltin oxide preparation) or a few crystals of menthol or thymol are placed to inhibit bacterial action. This is important because the bacterial growth causes breakdown of the proteinaceous material with release of ammonia, causing the urine to become more alkaline. Under alkaline conditions, the resin expands and the columns become badly clogged.

The pH of a measured amount of the collected urine is adjusted to 4.5, and the urine is fed into the resin column by the automatic feeding system. The flow of urine through the resin is controlled so that it is no faster than one drop every three seconds. With our apparatus, this is about 2 ml per minute. With large amounts of lead in urine, we find this slow rate to be necessary for complete extraction of lead. A liter will pass through in eight to nine hours.

Some of the colored material of the urine clings to the resin. It is removed with 400 ml of 1:3 solution of acetone and water. Then the resin is washed with 600 ml of deionized water. It may not be necessary to use so large a volume since most of the colored material comes out in the first several ml of acetone wash. However, we find that if the lead analysis is made on the polarograph, the half-wave potential is better defined if as much of the coloring matter as possible is eliminated.

The urine and wash are discarded and the

lead is eluted from the resin with 10 ml of 2N HCl followed by 10 ml of deionized water. If the HCl is dripped through very slowly, this is sufficient for almost complete lead recovery (200 gammas is recovered to 90% or better).

The 20 ml of 1N HCl is now analyzed for lead directly if the dithizone method is used, or polarographically if the amount of lead is sufficient to measure. On the Sargent XXI, we find two or three gammas of lead per ml is detectable, 5 gammas/ml is measurable.

If the lead in the 20 ml of 1N HCl is less than 80 or 100 gammas, the solution is evaporated to dryness in the beaker in which it was collected. We use an infrared lamp for this purpose. The gray film is redissolved in one or two ml of 1N HCl (0.5 ml may be used.) After complete solution, this is poured into the cell of the polarograph for measurement. As the polarograph measures concentration and the standard curve obtained is plotted in γ /ml, no rinsing of the beaker is necessary but solution should be complete and homogeneous.

The lead concentration is then calculated from a standard curve and the amount of urine used.

Results

Experiments were done to establish: (1) the completeness of extraction of lead from urine by the resin; (2) the completeness of elution of lead from the resin column by HCl; and (3) that the cation eluted from the resin column and showing a half-wave potential at about -0.44 volts in 1N HCl is lead.

The urine after having passed through the resin column with the adopted procedure was evaporated and acid digested and this material tested for lead by the sensitive dithizone method. No lead was found. The water and acetone washes also were tested by the dithizone method and no lead was detected.

The Rohm and Haas⁸ company state that any adsorbed cation on Amberlite IRC-50 can be desorbed easily with dilute acid with regeneration efficiency approaching 100 per cent. In our own work, we find lead in lead spiked urine can be eluted completely, and with the use of 10 ml of 2N HCl and 10 ml of deionized water is recovered to the extent of 90 to 100 per cent, depending on the rate of dripping and the completeness of draining the last amount of liquid from the resin.

The dithizone method and the polarographic method give the same results for material eluted from the resin.

Urine of the "pooled variety" ("normals")

gave results varying from 10 to 40 micrograms per liter, the lower results in the warm months and the higher results in the cold months, as might be expected from the work of Kehoe.⁹ Several analyses of the same pool gave results that varied by only 2 or 3 micrograms per liter.

TABLE I
Lead Content of Urine (Pooled) of Normal Males
(500 ml used for analysis)

Sample No.	Lead Content in Micrograms per Liter Urine	
	Resin plus polarograph	Resin plus dithizone
1	11; 14	12
2	15	12
3	34	
4	14	
5	10	
6	34	
7	38	
8	40	

TABLE II
Lead Recoveries from Spiked Urine
(500 ml used for analysis)

Urine Pool. No.	Micrograms lead added	Micrograms lead recovered
1	0	17
	100	122
	200	214
2	0	7
	50	56, 56.5
	100	96
	200	186

TABLE III
Lead Content of Urine Samples from Exposed Humans

Sample No.	Lead Content in Micrograms per Liter Urine	
	Resin plus polarograph	Wet ashing or co-precipitation plus dithizone
1	420	380
2	80	60
3	50	40
4	43	30
5	28	20
6	238	200
7	245	227
8	289	238

Note: The dithizone results were obtained independently by cooperating laboratories.

Tables I, II, and III show the results of our analyses on pooled normal urine samples, on spiked normal urines, and on urine samples from persons suspected of having been exposed to toxic amounts of lead. Our lead determinations were done polarographically, but some dithizone results are shown for comparison. Table III shows the results obtained on the same urine sampled by using wet ashing and precipitation methods followed by dithizone determination, these values having been obtained not by us but rather by the organizations concerned with the human exposures.

It is noted that the lead determinations by the ion-exchange method are about 10% higher for the higher values and 30 to 40% higher for the lower values than those of the known methods. It is suggested that the ion-exchange method is inherently more accurate because the very small amount of handling of the samples prevents the obvious losses entailed in many manipulations. Also one is not bothered by losses of lead which occur with the precipitation method, nor by volatilization of lead such as might occur in acid digestion.

Comments

We have not yet established how long the resin may be used. At this time we have used the resin for more than 12 analyses (8 to 10 liters of urine) with no apparent deterioration. When the resin has become darkened, it can be regenerated to some extent with HNO_3 . This has not been sufficiently explored.

For urine, a volume of 500 ml to 1000 ml is a reasonable amount to use for normal lead values. The amount of lead is then 5 to 40 gammas.

Mention should also be made of the fact that we find on the polarograms of eluted material a half-wave potential other than that of lead, which we believe to be due to copper. It occurs ahead of the lead wave and in the quantities we have found does not interfere with the lead determination. We have not yet established if copper is extracted quantitatively under the same conditions as lead.

Work presently under way may give an answer to the question whether EDTA used in therapy of lead exposure cases causes interferences in the present method.

Summary

The work described was limited to the determination of lead in urine by concentrating lead on an ion-exchange resin, eluting the lead from the resin with acid and determination of the lead by polarography or by the dithizone

method. The possibility is suggested that ion-exchange concentration and subsequent elution of elements can play a vital role in extending trace element techniques in the field of biochemistry, industrial hygiene, and air pollution. We expect to investigate this method for the determination of such elements as thallium and nickel which are of interest to us as industrial hazards.

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AMERICAN MUSEUM OF HEALTH

AN AMERICAN MUSEUM OF HEALTH has been granted a Charter, as an educational institution, by the Board of Regents of the University of the State of New York. It is planned that the newly-created institution will erect a \$3.5 million Hall of Medicine and Public Health at the World's Fair 1964-65 in New York City. In view of the national significance of the Museum and in recognition of the public interest that will be served by a medical and health pavilion, Fair officials have agreed that 70,000 square feet of space will be made available in the special exhibits area of the Fair. The pavilion will be the principal agency for medical and health exhibits at the Fair, and will be developed with the view of establishing a permanent Health Museum in New York City prior to the termination of the Fair. The Museum will have the only building at the Fair containing a comprehensive treatment of health information, health education, and the relationship of medical and companion sciences to man and himself.

The theme to be presented in the Museum is the FAMILY, which is the basic unit of our society. The specific programs or exhibits have not been designated but under study are the following general areas: man, medicine, public health, dentistry, nursing, community facilities and services, water supply, sewage disposal and sanitation, exercise, recreation, and special subjects and services. (We sincerely trust that the field of industrial hygiene and occupational health will receive appropriate recognition and coverage in this undertaking.)

During the New York World's Fair of 1939-40 the exhibits in the Medicine and Public Health Building attracted 11,800,000 visitors making this one of the ten most popular exhibits at the Fair. It is estimated that the proposed American Museum of Health will afford an estimated 20,000,000 visitors an opportunity to acquire useful knowledge concerning the human body from the prenatal period to old age. Funds for this large scale project are being received from insurance companies, pharmaceutical and other business groups, government agencies, private foundations, and individuals. For additional information please refer to—Dr. Howard R. Craig, Director, The New York Academy of Medicine, 2 East 103 Street, New York 29, New York.

The Differential Thermal Analysis of Quartz

DOUGLAS K. CRAIG, M.S.*

Dust and Ventilation Division, Transvaal and Orange Free State Chamber of Mines Research Laboratory, South Africa

The differential thermal method for the analysis of quartz is discussed and the literature briefly reviewed. The design and construction of a D.T.A. apparatus is described, and details of some of the results obtained with the present apparatus are given.

Introduction

CRYSTALLINE quartz has been proven considerably more toxic than the other constituents of dust that occur in mine air. It is therefore of importance to know not only how much dust there is in the air that the miners breathe, but also how much of that dust is dangerous; that is, consists of quartz particles.

In the gold mines of the Transvaal and Orange Free State (South Africa) it is desirable to know how the quartz content of the respirable airborne dust varies from mine to mine, from place to place in one mine, and even from one dust generating mining operation to another. Methods of assessing the quantity of crystalline silica in a dust sample depend on the physical and chemical properties of the quartz. They are such that they can, in general, be carried out only by laboratories that are especially equipped for the purpose, and not by the normal ventilation department on a mine.

The x-ray diffraction technique for the determination of quartz has been used in the Research Laboratories of the Transvaal and Orange Free State Chamber of Mines for some years, but it has not been possible to check the results of the analyses by any but the tedious and uncertain chemical methods. An attempt has therefore been made to apply the method of Differential Thermal Analysis (hereafter referred to as D.T.A.) to the determination of the quartz content of dust samples.

Principles of the D.T.A. Method

D.T.A. makes use of the fact that when a substance is heated it may undergo one or more

of several physical and chemical changes. These are usually accompanied by the evolution (exothermic) or absorption (endothermic) of heat. Both the temperature at which such a reaction occurs and the amount of heat involved are characteristic of the substance being treated. These heat effects can be used for both qualitative and quantitative identification of minerals in samples.

The term "quartz" is reserved in this paper for crystalline free silica (SiO_2). This substance displays a small, but sharp, reversible thermal change associated with the latent heat of inversion of α and β quartz at about 573°C , and it is this that is used in the D.T.A. method for the estimation of quartz.

The dust sample to be analysed and an inert powder (one that displays no thermal reactions in the temperature range of interest) are packed into two identical sample holders. A differential thermocouple (Figure 1) is introduced into these holders, which are mounted so as to be located at a fixed point inside an electrical furnace, inside which the temperature distribution should be as uniform as possible. A separate thermocouple is used to record the temperature of the furnace, which is heated up at a suitable rate, usually between 5 and 20°C per minute. The unknown and the reference sample are at the same temperature until some thermal reaction takes place in the former, when its temperature either rises above or falls below that of the inert sample. On heating, the α to β crystal inversion of quartz gives rise to an endothermic reaction.

This temperature difference causes a minute potential to develop across the differential thermocouple, its magnitude depending on this difference. The above potential has to be amplified to a suitable level for accurate recording or observation. The magnitude of the thermal effect is measured by either the peak height of the deflection from the base line or more commonly by the area under the peak. Calibration

* Formerly Physicist, Transvaal and Orange Free State Chamber of Mines Research Laboratory, Dust and Ventilation Division, now Health Physicist, South African Atomic Energy Board and at present attached to the Department of Radiation Biology, University of Rochester, Rochester, N. Y.

This paper describes a portion of the work which was presented in thesis form to the University of the Witwatersrand in partial fulfillment of the requirements for the degree of Master of Science.

tests involving samples of known quartz content enable these readings to be related to the percentage of quartz in the test sample.

Previous Work

D.T.A. has been widely used, especially in mineralogy and in the ceramics industry. Only attempts to apply the method to the study of quartz will be considered here. Details of the historical development of the technique may be found in several textbooks and reviews devoted to D.T.A.^{1, 2, 3, 4} The first work on quartz to be described in any detail was that of Berkelhamer^{5, 6} in 1944. One paper concerns the design and construction of the apparatus he used, while the second describes his work on quartz.

Several other workers have published results of investigations on the D.T.A. of quartz.⁷⁻¹⁸ Clelland, Cumming, Dempster and Ritchie¹⁹⁻²³ in a series of papers written separately and collectively discuss the presence of a high-solubility layer on the surface of finely-ground quartz, its removal by hydrofluoric acid etching, and its regeneration by purely mechanical grinding. This point is taken up by Nagelschmidt²⁴ with respect to the x-ray diffraction analysis of finely-ground quartz particles before and after etching.

Lack of space prevents a detailed description of the results obtained by various workers to be given here, but the point that stands out is that many of them arrive at contradictory conclusions. Grimshaw and Roberts¹⁰ state that the magnitude of the thermal response is not influenced by the environment of the quartz, and obtained good agreement between the percentage of crystalline quartz in samples of quartz rock from different sources as determined by D.T.A. and the Trostel and Wynne chemical method.²⁵ Other workers report anomalous results for D.T.A. tests on supposedly pure quartz samples from widely differing sources.¹²⁻¹⁶ Many report that the D.T.A. response is independent of particle size,^{6, 7} while others state that the effect decreases with particle size.¹¹

In summarizing the present status of D.T.A. as an analytical tool in quartz investigations, Grimshaw and Roberts² emphasize the need for the strictest possible standardization of technique for quantitative analysis.

A study of the literature relating to absolute quartz determinations has revealed a very real need for a further investigation of such determinations. Much of the work that has been published has given only very inadequate descriptions of the techniques used for making quartz assessments. Some factors that seem to the present author to be fundamentally impor-



FIGURE 1. The differential thermocouple is shown diagrammatically. The line of dashes indicates platinum -10% rhodium; the dots indicate the palladium -40% gold side of the couple. The test sample is placed in "a" and the inert sample in "b". V_T represents the potential due to temperature difference T .

tant in quantitative D.T.A. appear to have been completely disregarded by most workers. This paper will describe some of the work that has been carried out in the Research Laboratories of the Transvaal and Orange Free State Chamber of Mines.

D.T.A. Apparatus

Requirements of the Project

The requirements of a D.T.A. apparatus to be applied specifically to quartz determinations are somewhat different from those of one designed for more general work. Firstly, only the temperature range from 500 to 650°C is of concern; secondly, the heat of the quartz reaction is very small, being of the order of three calories per gram of active material, and giving rise under the most favourable conditions to a maximum temperature differential of about 3°C in contrast to differences of up to 80°C for substances like calcium chloride; thirdly, large samples of respirable dust take a long time to collect, so the apparatus must be designed to handle small samples.

The specific requirements of the D.T.A. apparatus for this project are therefore: (a) a highly reproducible, but not necessarily linear, rate of temperature change in the range from 500 to 650°C; (b) equipment sensitive enough to measure 5% of quartz in a sample, that is, a temperature difference between the two sample holders of the order of 0.15°C; and (c) the sample holders must be designed to admit less than about 300 mg of dust. Most previous workers have used sample weights of from 500 to 1500 mg.

In addition, there are the more general requirements that the equipment should be robust and easy to operate, and should give a highly reproducible response that is easy to assess.

Design of the Apparatus Used in This Laboratory

A D.T.A. apparatus usually consists of four main components: (a) an electrical muffle furnace; (b) a temperature program controller; (c) the sample holders and differential thermocouples; and (d) the differential temperature recorder.

Satisfactory results have been reported for widely differing types of equipment, and careful control of experimental techniques is probably of more significance in determining the accuracy of results.

Only a brief description of the present apparatus can be given here. Two 2000-watt cylindrical furnaces were constructed, the one mounted horizontally and the other vertically. The sample holders consisted of twin platinum crucibles for the former and a nickel block with two wells sunk into it for the latter, the interior dimensions of the wells and crucibles being identical. "Pallaplat" differential thermocouples (Pd-40% Au versus Pt-10% Rh) were used in both cases, the furnace temperatures being indicated by separate Pt versus Pt-13% Rh thermocouples.

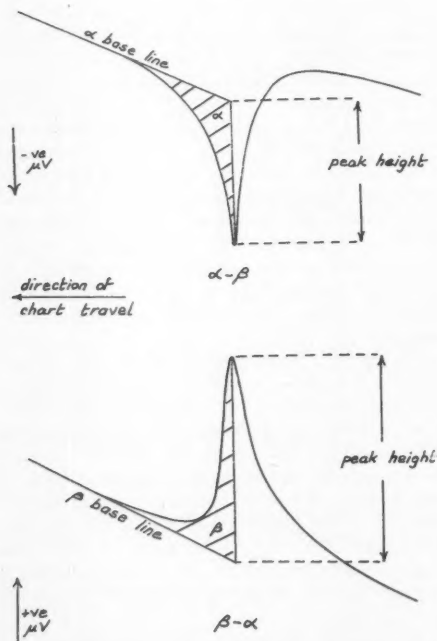


FIGURE 2. Assessment of D.T.A. response: method finally adopted.

A constant power input into the electrical windings of the respective furnaces is used to achieve a reproducible heating cycle. The differential temperature recording is effected by means of a Leeds and Northrup stabilized D.C. microvolt amplifier feeding into a Sunvic 10 mV potentiometer recorder. A centre-tap potentiometer device enables the recorder zero to be set at any point on the chart scale irrespective of the input from the amplifier.

The performance of this equipment has been found most satisfactory, the furnace heating cycles being accurately reproducible and the baseline drift with inert dust in both sample holders completely negligible. A temperature differential of only 0.02°C between the test and inert samples gives an output of one μV from the differential thermocouples, and a deflection of two small divisions (0.2 in.) on the recorder. This can be read to $\frac{1}{2}$ of a division, or 0.002°C .

Experimentally, a pure quartz test sample gives a potential of 125 μV in the horizontal and 75 μV in the vertical furnace under roughly the same conditions. Thus on the most sensitive amplifier range (50 μV full-scale) it should be possible to detect as little as 1% quartz quite easily. Whether or not this amount of quartz would yield reproducible results is another question.

Experimental Work

Techniques

Only a few of the more important observations will be presented. Particular attention was paid to the careful standardization of the methods used for obtaining thermograms in the present work. The same inert dust (minus 270 mesh calcined alumina) was used for all tests, and standard methods were adopted for the preparation and packing of both this and the test sample.

Many different methods have been employed for assessing the magnitude of the D.T.A. response. Webb²³ has made a comprehensive survey of these, and points out that they all appear to be somewhat arbitrary, except in the rare event of the base line before and after a peak being horizontal and on the same level. Due to the change in specific heat and thermal conductivity of the sample as it changes from the α to the β crystalline structure, this never occurs with quartz.

After analysing the results of several methods for measuring the peak height and the peak area of the response, that illustrated in Figure 2 was adopted. The centre line from the apex of the

peak and perpendicular to the direction of chart travel is drawn. As the peak is usually very sharp, this point is easily determined. The base line preceding the peak is usually very stable, and this is produced to intersect the centre line. The area and height to be measured are indicated.

Most workers claim that it is the area, and not the height, that is a measure of the heat involved in the reaction, but Grimshaw and Roberts¹⁰ report a linear relationship between peak height and percentage quartz.

There is considerable theoretical justification for assessing the response in the way chosen, firstly because the sloping base line is caused by the change in specific heat of quartz with temperature, and secondly because the heat reaction is actually complete before the return of the curve to its new base line. Both peak height and peak area measurements were made on all thermograms in the present work.

Preliminary measurements on a pure quartz sample yielded some interesting results. Firstly, height measurements somewhat surprisingly showed better reproducibility than the corresponding areas. For the same packing of the sample the variance was less than 2%, whereas for the areas it went up to 11%.

The variances for the horizontal furnace measurements were less than those for the vertical furnace. This is surprising, in view of the claims that have been made for nickel block sample holders in the literature. The response from the platinum crucibles was about 1.5 times that of the nickel block under the conditions of operation for the respective furnaces.

Thirdly, the statistical calculations carried out on these figures showed that between-packing differences could not be accounted for by random experimental variation alone. This finding seems to be of considerable importance since it appears to have been neglected by other workers in this field.

Finally, as successive runs on the same packing of a sample do not show any tendency towards a lower D.T.A. response, it is extremely unlikely that some of the β quartz does not revert to α quartz on cooling.

Results from the two furnaces showed the same trends throughout, and for the most part only those for the platinum crucibles in the horizontal furnace will be given.

Effect of Variables on the D.T.A. Response

The variables that are considered in this section all have an influence on the magnitude of the D.T.A. response, and several of them are

interdependent. For example, the mass of a sample that can be packed into the holders depends on the constituents of the dust and on its particle size distribution, and both these factors influence the thermal conductivity of the test sample.

(A) Packing of the test sample.

This is probably the most important of the variables investigated, not so much on its own account as on its dependence and influence on other factors. Within-sample variations in mass, the density of sample packing and the variation in mass between samples have to be considered.

Any variation in response due to repacking of the inert material will form part of that obtained with repacking of the test sample. Only the effect of the latter was considered in this work. Even using a standard technique, mass varied widely from one packing to another. Using a hand-packing method, differences in sample mass of the order of 12% commonly occurred, and the between-packing differences in the thermal response were larger than could be accounted for by random experimental variation.

On the basis of these results a constant pressure packing device was designed. This has enabled the masses of duplicate packings of a sample to be reproduced to within 2%, but only became available after much of the present work had been completed.

In the past, various workers have used widely differing methods of packing their test samples. The effect of varying the hardness of packing a dust into the holders was tested by conducting three cycles on each of five different packings, masses of approximately 200, 220, 240, 260, and 280 mg being packed into the sample holders to just fill them in each case.

The variation in D.T.A. response with mass was linear, but only 60% of the mass variation. The change in response is therefore a function of some other factor in addition to sample mass.

A more serious matter is the variation in sample mass (using a standard technique) from one sample to another. This increases by 70%, for example, as the average particle size of quartz dust increases from $<0.5 \mu$ to $>5.0 \mu$, and for a series of samples collected in mines by electrostatic precipitation, masses were found to vary from 178 to 273 mg.

Attempts to construct an appropriate correction curve for sample mass on the basis of the x-ray diffraction analyses for quartz on a number of samples have so far failed. This matter has received scant attention in the literature. Some workers quote their results as "per unit mass" but give no indication as to how they

carried out the correction or what justification they had for making such a correction.

The particle size distribution and thermal conductivity of the sample enter into this mat-

TABLE I
Results of D.T.A. and X-ray Tests on
Sized Fractions of Quartz

Sample No.	Description of Sample	Before/After HF Treatment	Mass of Sample in mg	D.T.A. Response				% Quartz by X-ray
				$\alpha \rightarrow \beta$		$\beta \rightarrow \alpha$		
				Height	Area	Height	Area	
15	<0.5 μ fraction	Before	154	28.2	12.89	31.2	14.09	72
		After	194	41.0	20.17	44.7	20.92	80
16	0.5-2 μ fraction	Before	192	40.2	18.19	48.2	20.51	82
		After	242	52.6	26.53	64.0	28.44	87
17	2-5 μ fraction	Before	224	82.0	41.37	94.2	39.28	98
		After	273	95.6	45.85	112.4	45.03	102
18	>5 μ fraction	Before	264	103.8	49.57	116.2	44.24	104
		After	282	114.4	52.44	125.4	51.90	107
19	Original D. & D. Dust	Before	214	75.8	32.30	93.0	38.29	94
		After	285	97.8	46.29	112.0	48.41	106

TABLE II
Percentage Silica Lost During
HF Etching Treatment

Sample No.	15 <0.5 μ	16 0.5-2 μ	17 2-5 μ	18 >5 μ	19 Original
Average % SiO_2 lost	25	14	9	8	9

ter, and further carefully planned D.T.A. tests are indicated. In the work that follows, no allowance has been made for sample mass even though this was measured in each case.

(B) Particle size distribution of dust in samples.

D.T.A. tests were conducted on various sized fractions of quartz prepared from respirable dust. The samples were microscopically sized and were analysed by both D.T.A. and x-ray diffraction techniques before and after etching for ten minutes with hydrofluoric acid. This treatment resulted in a slight decrease of the average particle size of the fractions.

The results of these tests are presented in Table I, peak heights being measured in microvolts and peak areas in microvolt-minutes.

It is apparent that the D.T.A. response drops very considerably as the average particle size of

the sample decreases, as does the sample mass. The x-ray results are also affected, and the standard appears not to be pure quartz.

In order to test the premise that there might be a higher percentage (per unit mass) of high-solubility material present on the surface of the smaller particles, the average percentage silica lost during the HF treatment was measured. The results are given in Table II. Microscopic examination revealed that the treatment resulted in a shift toward smaller sizes in the particle size distribution, not a disappearance of the smaller particles. If the change in sample mass is taken into account, then the change in D.T.A. response following the etching appears to be negligible for all but the smallest size fraction.

(C) Etching of quartz samples with 4% hydrofluoric acid.

The variation in sample mass and D.T.A. response of samples of similar total silica content rendered the construction of reliable calibration curves for the equipment difficult. For five such samples the one which yielded the highest D.T.A. effect gave the lowest x-ray response.

A series of etching tests were therefore carried out on a sample of superfine quartz dust (No. 10) that was used for animal inhalation experiments. This dust was etched for varying lengths of time, and then analysed by both D.T.A. and x-ray diffraction. The results are given in Table III, together with those for the sample which had previously yielded the highest D.T.A. response (No. 1).

Microscopic sizing of the samples showed that the HF treatment tends to reduce the particle size. After 25 minutes of etching, however, there was a sudden decrease in the number of very small particles. The percentage silica lost by the treatment also increased suddenly. This seems to indicate that the initial etching may cause the loss of a highly soluble layer of silica, but that increasing the etching time beyond 20 minutes might lead to the dissolving of the smaller quartz particles.

(D) Thermal conductivity of test sample.

The magnitude of the differential thermal peaks obtained from a given sample depends not only on the quantity of heat developed (or absorbed) during a reaction but also upon the rate at which that heat is transferred to or from the thermocouple junctions.

The areas under a D.T.A. reaction peak is proportional to HD/C , where H is the heat of reaction per unit mass, D is the bulk density and C is the thermal conductivity of the test sample. Hence test samples of high thermal conductivity may be expected to give lower D.T.A. responses than ones with low conductivity even if H and

TABLE III
Effect of Hydrofluoric Acid Etching on D.T.A. and X-ray Responses

Sample No.	Description of Sample	Mass of Sample in mg.	D.T.A. Response				% SiO ₂ lost in etching	% SiO ₂ by X- ray
			$\alpha \rightarrow \beta$		$\beta \rightarrow \alpha$			
			Height	Area	Height	Area		
1	Minus 325 mesh ground quartz crystal	273	114.4	47.57	129.6	44.95	—	87
10	Superfine D. & D. quartz dust	202	65.8	31.2	77.8	31.5	—	92
31	No. 10 etched for 5 minutes	276	88.0	41.09	105.0	45.85	14.3	102
32	No. 10 etched for 10 minutes	273	95.2	38.36	112.2	46.15	22.2	101
33	No. 10 etched for 15 minutes	297	101.0	51.93	113.0	48.55	22.8	95
34	No. 10 etched for 20 minutes	271	97.4	48.20	112.8	43.18	23.4	98
35	No. 10 etched for 25 minutes	280	100.0	44.38	114.0	46.09	27.2	99

D, which depends on particle size and packing, remain the same.

The results presented so far indicate the possible importance of thermal conductivity on the D.T.A. response in so far as this varies with density of packing, particle size and the nature of contaminants of the test sample. It is possible that it is differences in thermal conductivity which give rise to lower quartz results by D.T.A. than by the x-ray method for small particle sizes.

A certain amount of experimental work has been conducted on a technique of diluting every test sample with 75% of the standard inert dust in order to obtain samples of more uniform conductivity. This has not proved to be successful as both the D.T.A. response and the variation in response between quartz samples are uniformly reduced to one quarter of their previous value.

(E) Other variables.

The effect of other factors such as pre-calcination of the test sample, contaminants in the test sample, and changes in the heating rate have been considered, and a certain amount of experimental work conducted. However, none of these are considered likely to be responsible for the observed discrepancies between D.T.A. and x-ray results. They will, therefore, not be described in this paper.

Calibration tests

The purpose of these tests was to examine the linearity of the D.T.A. response for the various parameters measured and to construct calibration curves for the apparatus by means of which the percentage of quartz in an unknown test

sample could be estimated. Superfine "Dowson and Dobson" quartz dust was chosen as the standard, and using minus 270 mesh calcined alumina as the diluent, samples containing 80, 60, 40 and 20 per cent by weight were prepared. Two cycles on each of four packings were carried out for both furnaces on all these five samples.

The averages of the observations are given in Table IV, where the height measurements are in microvolts and the areas in microvolt minutes. No mass corrections have been made, although it is obvious that the low sample mass of the 100% quartz response has an effect, especially on the vertical furnace results. For both furnaces and both inversions a straight line has been fitted to the area and a parabola to the height measurements. These curves are all found to fit the observations satisfactorily, and Figure 3 serves as an example.

To these curves 95% confidence intervals have been fitted on the basis of the individual readings from which the average response at each point was obtained. The average coefficients of variation obtained are given in Table V. These limits are very stringent as they apply to a single observation on a particular sample. Duplication of both packing and cycles per packing would considerably increase the accuracy of assessment of a sample.

Tests on Actual Dust Samples

All the data that has so far been presented in this paper has concerned "pure" quartz samples. A limited amount of work has been done on dust that has been collected in the mines, some being electrostatic precipitator samples and others

TABLE IV
Calibration Test Results

% of Sample # 10	Horizontal Furnace					Vertical Furnace					% SiO ₂ by X-ray	% Total SiO ₂ (chemical)
	Mass in mg	$\alpha \rightarrow \beta$		$\beta \rightarrow \alpha$		Mass in mg	$\alpha \rightarrow \beta$		$\beta \rightarrow \alpha$			
		Height	Area	Height	Area		Height	Area	Height	Area		
100	202	65.8	31.2	77.8	31.5	202	42.6	14.6	37.6	14.5	92.0	96.78
80	239	62.3	23.6	70.0	23.6	250	42.2	13.3	33.6	13.5	75.8	75.26
60	247	54.1	18.2	57.7	17.3	255	35.5	8.4	27.1	8.5	56.1	56.62
40	244	39.6	10.3	39.2	10.5	256	26.2	5.3	18.1	5.2	33.8	40.43
20	234	20.9	4.1	19.2	4.4	251	14.8	2.4	10.4	2.6	26.7	16.83

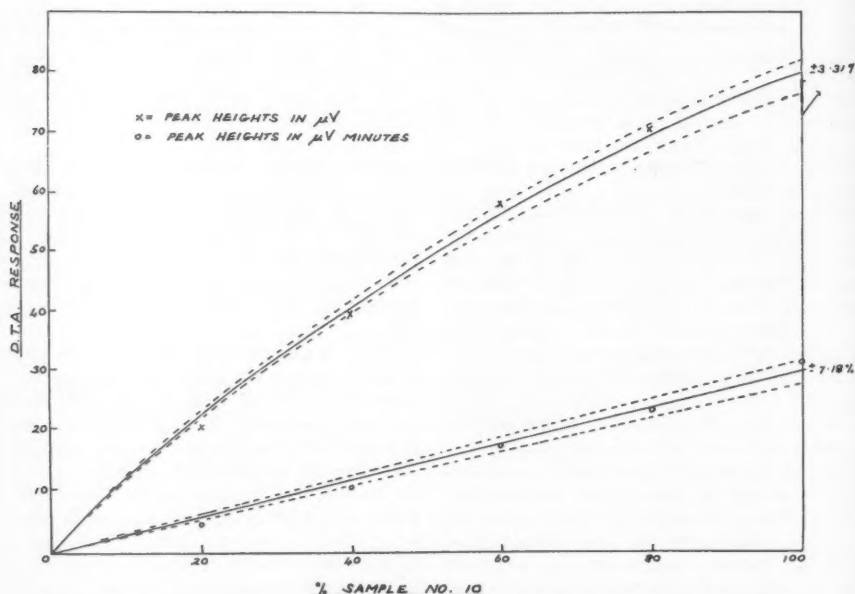


FIGURE 3. Showing the D.T.A. response vs the percentage of prepared sample for the $\beta \rightarrow \alpha$ inversion in the horizontal furnace.

TABLE V
Average Coefficients of Variation of Individual Readings from Statistical Curves Fitted to Calibration Readings

	$\alpha \rightarrow \beta$		$\beta \rightarrow \alpha$	
	Height %	Area %	Height %	Area %
Horizontal Furnace	± 2.24	± 6.58	± 3.31	± 7.18
Vertical Furnace	± 2.55	± 15.12	± 5.71	± 12.00

"grab" samples of non-airborne dust. The results have been discouraging in so far as the D.T.A. response indicated in general a quartz content that was less than half as great as was obtained on the same samples by x-ray diffraction analysis.

Discussion and Conclusions

The work conducted in this Laboratory to date has shown that while very small quantities of quartz in a dust sample can easily be detected, wide variations in response occur even with samples that are composed almost entirely

of quartz. This variation also occurs with x-ray diffraction analysis, and the correlation between determinations of quartz content in dust samples of unknown composition by the two methods has been found to be poor. The reproducibility of the D.T.A. results for a particular sample is quite good, more so for peak height than for peak area measurements.

Because of lack of space, discussion of these results must be curtailed to a minimum. However, several workers have reported the presence of a high-solubility layer on the surface of quartz particles,¹⁹⁻²² and have concluded that it is a vitreous silica layer of the Beilby type. It can be removed by etching the dust with hydrofluoric acid, and its removal increases rather than decreases the toxicity of the quartz dust.²⁰

The presence of such a non-crystalline silica layer would exert an increasing influence on the D.T.A. response as the average particle size of the dust decreased. Further investigations are necessary, but the results do show that neither the increase in sample mass nor the increase in D.T.A. response after etching can be attributed to a marked change in particle size distribution. The effect appears to be absent for particle size fractions larger than the ones tested in this work, but as they constitute non-respirable dust, they are of no interest to us.

It also seems possible that the thermal conductivity of a sample depends on the particle size distribution of the dust. The etching tests indicate that the high-solubility layer may be responsible in some way for this change, and the only solution seems to be to actually measure the conductivity of various size fractions of dust before and after etching. It has been reported that the non-crystalline silica layer is of roughly constant thickness²² regardless of particle size. However, electron microscope studies of quartz dust in this Laboratory have failed to confirm the presence of such a layer. This matter is as yet unresolved, and further research is needed.

Concerning the calibration tests, the fact that area measurements give a linear response is in agreement with theory, but some workers have claimed that peak heights yielded a linear calibration curve as well. This is definitely not the case in the present work which has been conducted on both heating and cooling cycles for two different types of sample holders. No previous attempts to fit higher order equations to peak height observations seem to have been made.

The objective of calibrating the apparatus for quantitative quartz determinations was not

achieved due to the great difficulty experienced in obtaining a 100 per cent pure sample of crystalline quartz. There are several possible explanations for the poor correlation between x-ray and D.T.A. results. Firstly, the variable factors discussed under experimental work undoubtedly affect the D.T.A. results, especially since the samples of unknown quartz content were of highly variable size distributions. Secondly, as suggested by Grimshaw,¹¹ the x-ray measurements may not be as reliable as an absolute standard as has been accepted by most D.T.A. workers in the past. The x-ray response falls off on samples with an average particle size greater than about 5 to 10 microns, so most samples collected by electrostatic precipitation have to be ground down before analysis, a procedure which might will affect the crystalline quartz content.²³ Thirdly, the possibility that quartz from different geological environments may have different latent heats of inversion cannot altogether be ruled out.

This latter factor might prove to be the ultimate limitation in using the D.T.A. method for quartz determinations. The author does not consider such variations to be responsible for the discrepancies reported in this paper, but thinks that the answer lies in more careful sample preparation before analysis. As we are specifically interested in the quartz content of respirable dust, it is felt that some method of sample preparations could be evolved whereby only the dust that does not settle out of a suspension in a fixed time interval (to be determined with respect to the lung retention curve) is analysed. Standard samples would be prepared in the same manner.

The author feels that further work along the lines indicated in this paper should render the D.T.A. method accurate and reliable for the determination of the percentage of quartz in samples of airborne mine dust.

Acknowledgments

Acknowledgement is due to the Transvaal and Orange Free State Chamber of Mines for permission to publish the results of the project, to Professor A. L. Hales and the late Professor H. J. Logie for their guidance and encouragement, and to my many colleagues on the staff of the Chamber of Mines Research Laboratories for their assistance.

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COURSES IN RADIOLOGICAL HEALTH

THE TWO-WEEK TRAINING COURSE, *Basic Radiological Health*, will be presented at the Robert A. Taft Sanitary Engineering Center, Cincinnati, Ohio, January 15-26, 1962, by the U. S. Public Health Service through its Division of Radiological Health. This course covers the basic nuclear physics necessary for a technical understanding of radiological problems in public health work, major sources of radiation exposure, modes of radiation injury, and units and terminology associated with this new field.

The course will be repeated at the Sanitary Engineering Center April 23 - May 4, 1962; at the Southwestern Radiological Health Laboratory, Las Vegas, Nevada, February 26-March 9, 1962; and at the Radiological Health Laboratory, Rockville, Maryland, May 7-18, 1962. A full description of the course is given in the *Training Program Bulletin* which is available upon request. Applications for the course or requests for information should be addressed to the Chief, Training Program, Robert A. Taft Sanitary Engineering Center, 4676 Columbia Parkway, Cincinnati 26, Ohio, or to a PHS Regional Office.

A Radiochemical Determination of Alpha Exposure from Enriched Uranium in Urine

JOHN S. ALERCIO, B.S., GEORGE A. WELFORD, M.S.,
and ROBERT S. MORSE

*Analytical Division, Health and Safety Laboratory, U.S. Atomic Energy
Commission, New York 14, New York*

A method is described for the separation and measurement of alpha activity from enriched uranium in urine. The uranium is collected by an alkaline phosphate precipitation and is isolated from urine constituents through ion exchange. The resulting solution is measured fluorimetrically for uranium and then electroplated for alpha count. Self absorption of the alpha particles is minimized by the preliminary ion exchange separation and by an improved electrodeposition technique. Fluorimetric recovery of added uranium from one liter of urine is approximately $95\% \pm 10\%$. The recovery by alpha count is $92\% \pm 10\%$.

Introduction

THE need for an accurate, efficient, relatively inexpensive and rapid method of measuring alpha activity from enriched uranium in urine of exposed personnel was indicated by the increased use of U^{235} in the Atomic Energy Program. While urine has been analyzed extensively by fluorimetric techniques¹ to determine quantitative exposure to normal uranium, little has been done to measure the exposure to alpha activity from enriched uranium.

The probable hazard from exposure to enriched uranium is its ionizing alpha radiation whereas permissible limits of exposure to natural uranium are based on its chemical toxicity. It has been established that the permissible concentration of natural uranium in urine is 50 micrograms per liter. This measurement can be made by fluorimetric analysis. The limit of detection by this method with the present techniques (Figure 1) is one microgram per liter. Alpha activity from one microgram of natural uranium is 1.51 disintegrations per minute, half from U^{238} and half from U^{234} . It follows that fifty micrograms of natural uranium shows the associated alpha activity of 75.5 disintegrations per minute. A typical enriched uranium which may be composed of one per cent of U^{234} , 93 per cent of U^{235} and six per cent of U^{238} shows an alpha activity of about 140 disintegrations per minute per microgram. This is almost twice the permissible concentration for alpha activity in urine based on the limit of sensitivity of the

fluorimetric analysis. Thus, it is obvious, that the fluorimetric method alone is not sensitive enough to determine alpha activity from enriched uranium in urine.

Numerous attempts have been made to make the procedure in urinalysis of enriched uranium more efficient by adding the electrodeposition and alpha counting techniques to chemical analysis.^{2,3} Among the efforts in this direction was the work of Hamrick.⁴ The major problem he encountered, as did others before him,⁵ was isolating the uranium from the bulk of other salts in the urine. Their poor total recovery of uranium resulted from inefficient electrodeposition. In the later efforts of Royster,⁶ better results were achieved because he partially separated the urine constituents from uranium by precipitating calcium as an oxalate and magnesium as a phosphate. Then, when Milligan and his group,⁷ and also Boni,⁸ introduced ion exchange to separate uranium from urine constituents, markedly better results became possible. From these efforts, it is clear that a good working procedure has to include a fluorimetric method, an efficient separation of uranium from urine constituents by ion exchange as well as an improved electrodeposition and alpha counting technique.⁹ Boni employs these criteria using the nuclear track technique for low level alpha count and the electronic count when levels are high enough.

In this article, the HASL procedure of measuring alpha activity from enriched uranium in urine based on refinements of existing fluori-



FIGURE 1. HASL uranium fluorimeter.



FIGURE 2. Ion exchange apparatus.

metric methods, ion exchange, electrodeposition and alpha counting techniques, is described.

Equipment and Reagents

APPARATUS

- (1) Soxhlet extractor, 2000 ml.
- (2) Ion exchange columns, 10 cm long and 1.4 cm inside diameter (Figure 2).
- (3) Separatory funnel, 500 ml.
- (4) Mercury cathode apparatus, Eberbach.
- (5) Uranium fluorimeter, HASL.
- (6) Nickel discs, 24 mm diameter, 0.032 inches thick.
- (7) Electroplating cell, teflon cylinder, 73 mm high and 38 mm outside diameter. Inside diameter on bottom (plating area)-18 mm. The cell tapers to an inside diameter at the top of 25 mm. A solid copper base is bored so that the dish and cylinder fit snugly. A top copper ring is bored through and a step shoulder is formed so that the cylinder fits snugly in place. The cell is held together by three copper rods screwed into the base at equal intervals and bolted with nuts through the top copper ring (Figure 3).
- (8) Platinum anode, platinum wire one mm in diameter and about 80 mm long with a 13-mm flat spiral wound bottom.
- (9) Electrolytic apparatus, Braun, Model PC (Figure 4).
- (10) Milliamp meter, 0-100 milliamps.



FIGURE 3. Electroplating cell assembly: at top from left to right are shown the copper base and supports, Teflon cylinder, nickel disc, copper ring (top), and assembly nuts. The complete assembled cell is shown at the bottom.

REAGENTS

All the reagents are Reagent Grade chemicals.

The resin used is Dowex 1 (4% DVB) 100-200 mesh, anion exchange.

Resin and Column Preparation

For resin preparation, extract 500 grams of Dowex 1 (4% DVB) (100-200 mesh) with one liter of methyl alcohol for approximately 24 hours in a Soxhlet extractor. Carry resin through a batchwise aqueous extraction with two liters of the following reagents:

- a) Distilled Water—(three washes, five minutes each).
- b) 1N NaOH—(½ hour wash).
- c) 3N NaOH—(½ hour wash).
- d) 1N HCl—(½ hour wash).
- e) 3N HCl—(½ hour wash).
- f) Distilled Water—(three washes, five minutes each).

Transfer 10 ml of the wet resin to the ion-exchange column. Pass 7N hydrochloric acid through the column until the effluent is 7N. About 500 ml of 7N HCl is necessary.

In this Laboratory the resin is not used again because of the danger of contamination in low-level samples. The resin is discarded and clean resin used for each sample.

REGENERATION OF RESIN BED

If resin is to be used again these steps of regeneration are necessary:

- (1) Backwash the column thoroughly with a large amount of distilled water (one liter).
- (2) Pass 200-250 ml of 1N HCl through the resin bed.
- (3) Then pass 7N HCl through the column until the effluent is 7N. About 500 ml of 7N HCl is necessary.
- (4) The column is now ready for the next sample pass.

Procedure

URANIUM COLLECTION BY ALKALINE PHOSPHATE PRECIPITATION

To one liter of a 24-hour urine specimen in a 1500-ml beaker add 20 ml of 10% phosphoric acid solution and 150 ml of concentrated nitric acid. Cover and boil gently for one hour. Allow to cool slightly and add 40% NaOH solution to precipitate the alkaline earths as phosphates (approx. pH 9). Add 10 ml excess. Digest on a steam bath with occasional stirring for one hour. Filter through Whatman #41 filter paper

by gravity and discard filtrate. Wash the precipitation beaker with 1:1 HNO_3 , at least four times with 25-ml portions pouring through the paper each time to dissolve the precipitate, and collect the solution in a 250-ml beaker. Wet ash the residue to a white salt by the addition of small amounts of nitric acid. Convert to chloride salts by the addition of two 5-ml portions of concentrated HCl and evaporate to dryness. Pick up with 100 ml or more of 7N HCl, until complete solution is effected, for passage of sample through a prepared resin column.

ISOLATION OF URANIUM WITH ION EXCHANGE RESIN

Transfer the solution to a 500 ml separatory funnel (Figure 2) and introduce to a prepared resin column at a flow rate of less than two ml per minute. Wash the resin with 400 ml of 9N HCl at a flow rate not exceeding two ml per minute. Elute the uranium from the resin with 200 ml of 1N HCl, collecting the eluate in a 250-ml beaker. Evaporate to dryness. Dissolve the residue in 50 ml of 0.3M sulfuric acid. The iron in the sample is removed by Hg cathode electrolysis. Electrolyze the sample for one hour at one ampere. Filter through #41 Whatman paper to separate the solution from the mercury and wash with distilled water. Evaporate the sample solution to dryness. Add five ml of concentrated HNO_3 and bring to dryness again. Dissolve the residue with 2-3 ml of concentrated HNO_3 . Heat gently and cool. Transfer the sample to a 10-ml volumetric flask and bring to volume with distilled water. Analyze for uranium fluorimetrically (Figure 1).

PREPARATION FOR PLATING AND DETERMINATION OF ALPHA ACTIVITY

Transfer the remainder of the sample from the fluorimetric analysis to a 50-ml beaker. Add five micrograms of normal uranium carrier from a standardized solution in 5% nitric acid, and evaporate the contents to dryness with low heat. Pick up the residue with two 10-ml portions and one 5-ml portion of 0.016M ammonium oxalate. After polishing beaker with each portion of ammonium oxalate, transfer to a prepared electroplating cell (Figure 3). (The cathode of the cell is a 24-mm nickel disc. The anode is a platinum electrode. The distance between the anode and the cathode is approx. 50 mm.) Add one ml of ethyl alcohol and electroplate at a current density of approx. 40 milliamps (Figure 4). The anode stirrer speed is approx. 200 rpm. The plating is done overnight

(17-20 hours) or until plating is complete. Test the electrolyte by fluorimetric analysis. When plating is complete, shut off current, and pour off supernatant liquid. Wash cell with distilled water. Dismantle cell. Wash disc with water and then ethyl alcohol. Flame disc to a dull red. Cool and mount using nylon ring and disc with alpha phosphor and transparent mylar (Figure 5). Make the alpha count in a scintillation



FIGURE 4. Electroplating apparatus.



FIGURE 5. Phosphor ring and disc assembly: shown from left to right in the top row are the nylon disc, electroplated source uranium, phosphor disc, transparent mylar, and nylon ring. The fully assembled ring and disc are shown at the bottom.

TABLE I
Recovery Data for Experiment No. 1

Sample #	Fluorimetric Recovery U^{235} micrograms			Activity Recovery U^{235} d/m		
	Added	Found	% Rec.	Added	Found	% Rec.
A	0.936 \pm 0.00	0.961	102.7	7.82 \pm 0.75	7.19	91.9
B		0.961	102.7		7.13	91.2
C		0.898	95.9		7.39	94.3
D		0.877	93.7		5.67	72.5
E	2.01 \pm 0.20	Lost		16.05 \pm 1.54	Lost	
F		1.98	98.5		14.53	90.5
G		1.68	83.6		11.33	70.6
H		1.98	98.5		14.99	93.4
I	3.30 \pm 0.06	3.00	90.9	22.69 \pm 0.30	22.39	98.7
J		2.90	87.9		18.99	83.7
K		2.90	87.9		20.99	92.5
L		2.90	87.9		20.99	101.3
Average % Recovery = 93.6 \pm 6.5%				Average % Recovery = 89.2 \pm 9.8%		

TABLE II
Recovery Data for Experiment No. 2

Sample #	Fluorimetric Recovery U^{235} micrograms			Activity Recovery U^{235} d/m		
	Added	Found	% Rec.	Added	Found	% Rec.
I	1.08 \pm 0.06	0.88	81.5	7.55 \pm 1.27	6.15	81.4
II		0.92	85.2		8.35	110.6
III		0.90	83.3		6.69	88.9
IV		0.92	85.2		Lost	
V	2.12 \pm 0.13	1.79	84.4	14.74 \pm 1.34	13.43	91.1
VI		1.99	93.9		14.89	101.0
VII		2.07	97.6		11.69	79.3
VIII		2.37	111.8		17.49	118.6
IX	3.18 \pm 0.13	3.28	103.1	19.63 \pm 3.2	17.41	88.7
X		3.49	109.7		18.99	96.7
XI		3.59	112.9		Lost	
XII		3.59	112.9		20.01	101.9
Average % Recovery = 96.8 \pm 12.8%				Average % Recovery = 95.8 \pm 12.5%		

counter with bare phototube. A 2 pi proportional counter may also be used for alpha counting.

Discussion

The complete separation of uranium from urine constituents is necessary for ideal electroplating. This indicates the importance of an efficient isolation method. In the method HASL developed, the calcium already present in urine is used as a carrier. To evaluate the recovery level of added uranium from one liter of urine, two separate experiments were run following

the above described procedure, using spikes of one, two and three micrograms of enriched uranium. Since the phosphor disc technique was not yet developed for these experiments, the uranium was plated on 29 mm diameter nickel discs and 2 pi counted. The activity is calculated from the formula:

$$d/m = (\text{net } c/m \times 2) - 7.55$$

with

c/m = counts per minute of a 30 or 60 minute count.

2 = 50% efficiency factor.

7.55 = d/m of 5 micrograms of added natural uranium.

Tables I and II present the recovery data for the two experiments. The recovery of uranium by fluorimetric analysis averages about 95% \pm 10% and recovery of alpha activity averages about 92% \pm 10%.

An estimate is made of the alpha exposure from enriched uranium by calculating the alpha activity from the fluorimetric recovery, based on normal uranium and subtracting that number from the total alpha activity found by actually plating and counting. No attempt has been made to determine the degree of enrichment of the uranium, since the important consideration is the degree of total alpha exposure.

Other techniques of preliminary collection of uranium from urine are being tried. These include ammonium hydroxide and organic reagents as the precipitants. The object of experimenting with ammonium hydroxide is to obtain smaller residues before ion exchange that are more soluble in 7N hydrochloric acid. But with the use of ammonium hydroxide the problem of greater carbonate complexing arises with ensuing loss of uranium.

The ion exchange technique is most essential for complete separation of uranium from urine constituents to achieve ideal electrodeposition of uranium. Therefore, careful attention is given this phase of the procedure.

The electroplating technique developed in this method has several distinct advantages because it gives an evenly distributed deposit on the plate with a very thin film that is desirable for alpha counting. Since plating is done with a very low current density it can be left unattended over night. There is no evaporation or bubbling of solution because no heat is required or produced in the cell.

While alpha counting can be done by conventional methods this laboratory has found the

phosphor disc technique⁶ more efficient. It protects against contamination, and maintains a very low background. By using added uranium, small amounts can be counted efficiently avoiding the tediousness of the nuclear track method which has the disadvantages of considering only a tenth of the total sample and of the possibility of subjective errors. In order to accommodate the phosphor disc alpha count technique the electroplating cell has been redesigned to take smaller nickel discs that can be directly mounted for alpha count.

Summary

In developing an efficient and accurate technique of analysis to determine exposure of working personnel to alpha activity from enriched uranium, existing methods have been modified and refined particularly as concerns the ion exchange, electroplating and alpha counting phases of this procedure. This laboratory can run about eighteen samples in three to four days with approximate accuracy of recovery of enriched uranium for a liter of urine at 95% \pm 10% by fluorimetric analysis and with an accuracy of about 92% \pm 10% in measurement of uranium by alpha count.

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A Double-wall Noise Control Enclosure for an Impulsive Sound Source

R. M. HOOVER, L. J. WILLIAMS, and W. G. RUSSERT

Bolt, Beranek and Newman, Inc., Cambridge, Massachusetts, and Linde Company, Division of Union Carbide Corporation, Indianapolis, Indiana

The design of a group of double-wall noise control enclosures on the upper floor of an existing building is described and some noise reduction measurements are reported. A design objective for these enclosures was to achieve more effective noise reduction than that provided by existing heavy, on-grade, double-wall, reverberant enclosures, but with reduced weight. This objective was achieved by a combination of a light weight inner skin, a moderately large air space, a masonry exterior, isolation of the inner room floor slab, and the use of sound absorbing materials. Other noise control measures included sound retarding doors and windows, mufflers in the air supply and return passages, and flexible service connections to the rooms. Peak and octave band noise levels taken inside and outside the old and new enclosures are reported.

Introduction

IN this paper we wish to review the design of a group of noise control enclosures or cubicles, provided for a facility doing development work on Linde's Flame-Plating process. We had the opportunity in this work to evaluate some existing enclosures before designing the new enclosures. A comparison of the old and new enclosures, both in design and effectiveness, will be presented. Also as the source was impulsive both octave band peak levels and octave band average levels are reported.

The basic design objective for the new enclosures, which had to be located on an upper floor of an existing building, was to achieve noise reduction at least as effective as that provided by the existing enclosures but with a reduction in weight. A secondary objective was to achieve even more effective noise reduction if this could be done within the existing weight limitations. To compare the new and old enclosures, the existing enclosures will be described first, and results of noise measurements at these enclosures will be reviewed.

Existing Enclosures

The noise source is a standard production type Linde Flame-Plating gun. In this patented device an oxy-acetylene mixture is detonated in a barrel to heat and propel suspended powder particles which are discharged from

this barrel at high velocity and against the surface to be coated. The barrel is several feet long and has a diameter of about one inch. The detonations are produced at a rate of four to five impacts per second.

The existing development facility, adjacent to a Flame-Plating plant of similar construction, consisted of a one-story concrete block building containing four enclosures and a central operating area. A plan view of two of the enclosures is shown in Figure 1. In each of these enclosures the gun, as shown in the drawing, was located inside a hard concrete room with dimensions of about $16 \times 12 \times 10$ ft. The wall construction consisted of a 90 lb/sq ft, 8-inch solid, concrete block inner wall, a 50 lb/sq ft 8-inch hollow, concrete block exterior wall, with an 8-inch air-space between. The inner wall and floor slab were supported on a 6-inch gravel base without any intended structural connections to the exterior wall. However, some rigid connections did exist via electrical conduit and framing around the double doors. The inner ceiling consisted of 8-inch precast concrete beams with an 18-inch airspace to the roof slab. The window consisted of a set of three $\frac{1}{4}$ inch plates in rubber gasketing in each wall. The doors consisted of an interior 4-inch solid concrete door, and an exterior 4-inch wood and cork door.

Noise levels were tape recorded both inside one of these enclosures and at various positions in the control room area. Both octave band peak and average levels measured inside an enclosure are shown in Figure 2. As indicated peak levels of about 150 db were recorded in several of the

R. M. Hoover and L. J. Williams are associated with Bolt, Beranek and Newman, Inc.; W. G. Russert is with Linde Company.

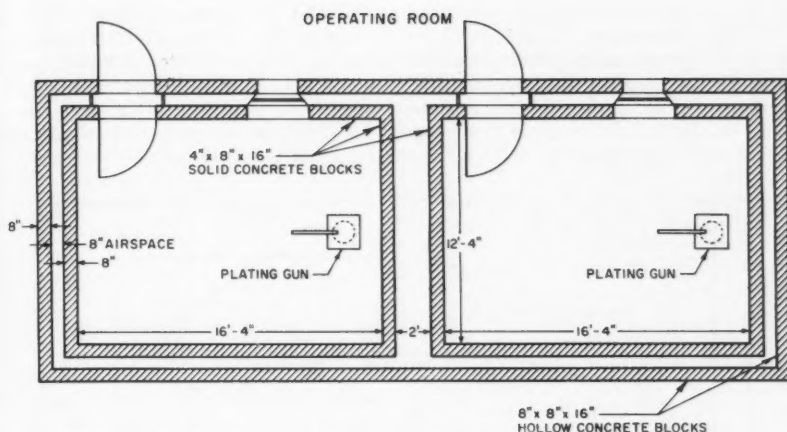


FIGURE 1. Plan diagram of the existing cubicles.

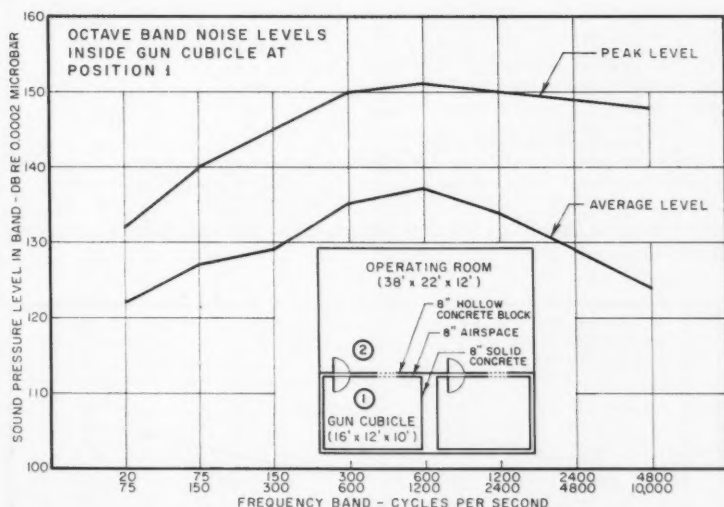


FIGURE 2. Noise level measurements at designated position (1) in the gun cubicle.

mid- and high-frequency octave bands. These peak levels were measured with a General Radio impact meter. The octave band average levels were obtained using the fast meter movement on a General Radio octave band analyzer. As indicated these octave band average levels ranged from about 120 db to 135 db.

Similar data which were measured in the control room about 4 feet from the wall midway between the door and window, are presented in Figure. 3. As shown octave band average levels of about 80 db were measured over most of the

frequency range. Other data were taken both near the window and near the door. These measurements indicated that the transmission through the door was the most controlling factor in the mid-frequency range. Although the seals provided were adequate it appeared that both the door panel and possibly the framing around the exterior door were limiting the effectiveness of the wall.

In Figure 4 the measured noise reduction (NR) of the existing enclosures based on both the peak and average data is shown by the

lower curve. The upper curve shows the *minimum* NR which it is estimated should have been achieved by the actual wall installation without any doors. This comparison is shown to indicate the degree by which the NR was reduced by the door installation. For example, the measured NR for this double masonry wall with the

8-inch airspace averaged about 13 db below the expected NR in the 4th, 5th and 6th octave bands. It should be noted, however, that an adequate NR of about 45 db was achieved in the first octave band, where the mean frequency is four times the lowest double-wall resonance frequency of about 10 cps.

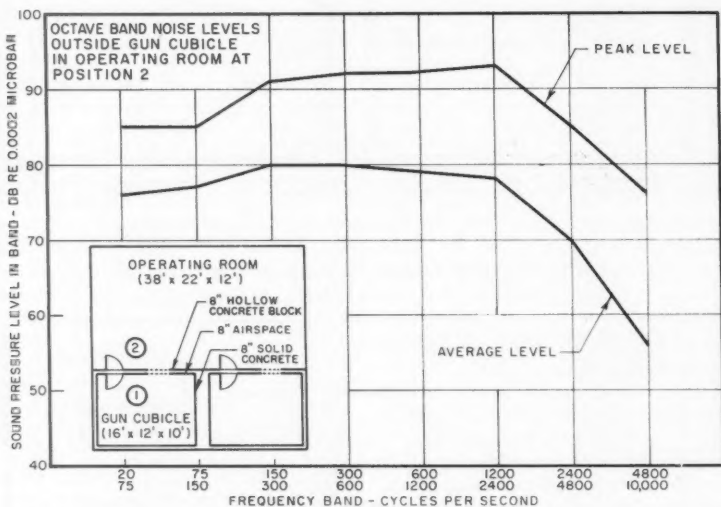


FIGURE 3. Noise level measurements at position (2) in the control room outside the gun cubicle.

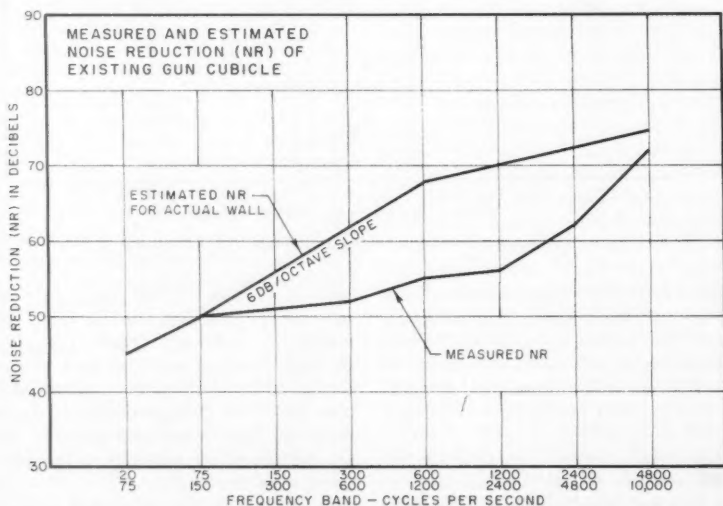


FIGURE 4. Showing the noise reduction (NR) estimated and measured for wall construction of existing cubicle.

To summarize, peak noise levels of up to 150 db, and average levels of up to 135 db were recorded inside an enclosure at the existing facility. In the control room average levels of up to 80 db were recorded with peak levels of over 90 db. The measured NR was found to be low for the construction due, in part, to the sound transmitted through the door.

New Enclosures

To design the new enclosures two basic factors were considered. First, as the new enclosures had to be on an upper floor of an existing building, the allowable added floor load for the enclosures was strictly limited. Secondly, although the noise levels in the control room of the existing facility were within accepted limits for safe working conditions it was recognized that some improvement in the control room noise environment could be achieved. A related factor was the desire to minimize the noise intruding to spaces below the facility. Considering these factors the NC 60-65 noise criteria curves were selected as a reasonable, but not critical, goal for the noise levels in the control room of the new facility.

The octave band NR required to achieve the NC 60-65 noise control criteria, compared with the measured NR of the existing facility is plotted in Figure 5. The figure illustrates two points of interest. First, in the 20-75 cps band it is seen that the NR of the new facility even

could be slightly less than that of the existing facility. As the NR with most materials in the 20-75 cps octave band is directly related to weight it was indicated that some weight reduction would be feasible in the new enclosures. Secondly, Figure 5 indicates the need for increasing the effective NR in the mid-frequency range by 15 to 20 db. While improving the door installation in the old facility would have achieved a large part of this additional NR, a balanced door design by itself, would not be necessarily adequate in the new enclosures with the weight limitations imposed by the existing structure.

From the NR requirements, namely 42 db in the 20-75 cps band and increasing at about 10 db/octave to about 75 db, it was determined that a double-wall enclosure was required for the new facility. However, to gain significant decrease in the weight of the required construction relative to the former construction, some measures not provided in the existing facility appeared necessary. It was decided to design the enclosure with a large airspace between the two walls, to achieve a low double-wall resonance, and to provide sound absorbing materials both inside the inner room and in the airspace between the two walls to control interior noise levels, and standing wave patterns between the walls. Also it was decided to isolate the inner room from the building by floating the floor. This isolation serves the purposes of providing

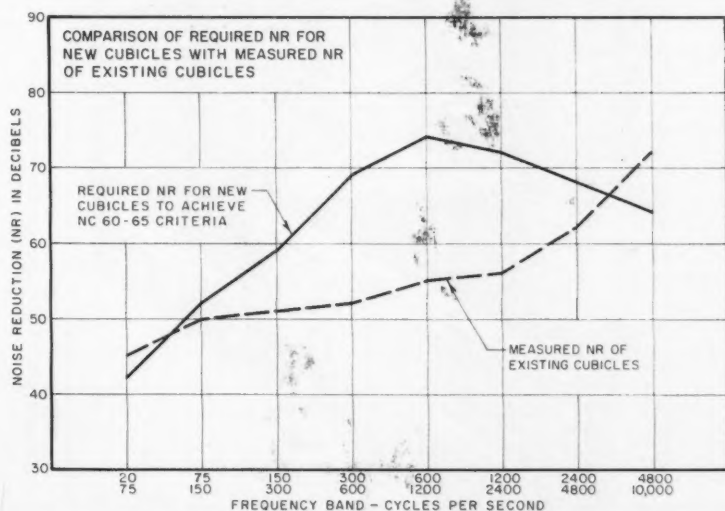


FIGURE 5. Showing the noise reduction (NR) required for the new cubicles as compared to the NR of existing cubicles.

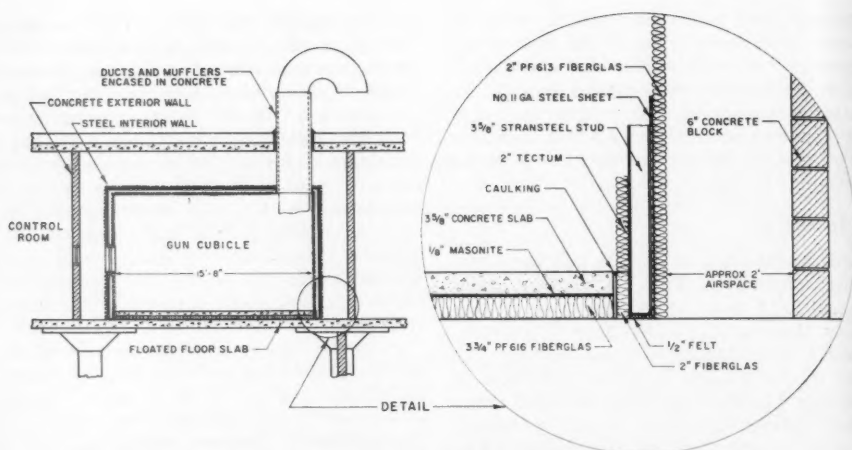


FIGURE 6. Section and detail drawings of the new cubicles.



FIGURE 7. View showing construction of outside wall window.

additional NR for airborne sound transmitted to spaces beneath, and also reduces the flanking of the double-wall structure.

With these considerations, the wall construc-

tion design finally selected, consisted of an 11 gauge steel panel inner wall, a 2-foot, airspace, and a 6-inch hollow concrete block outer wall as shown in Figure 6. Also in this figure the wall

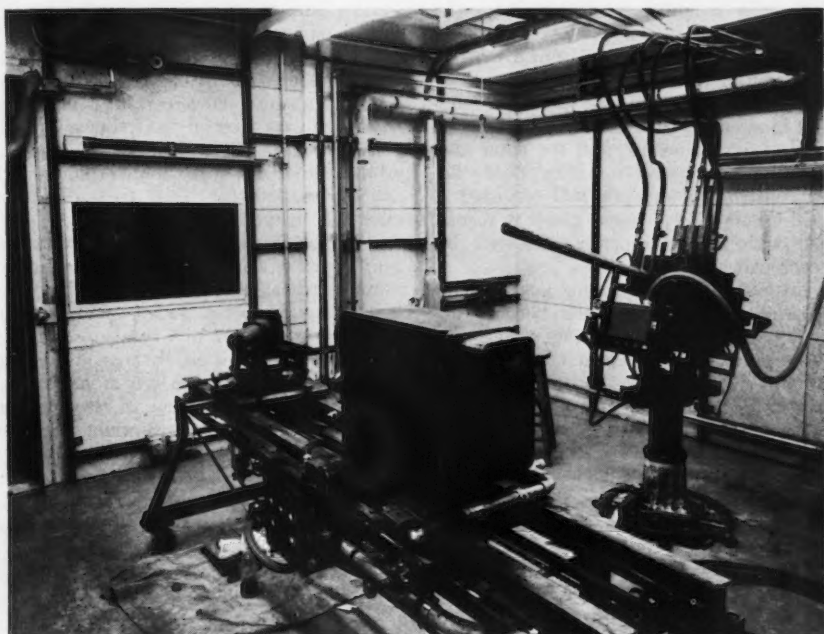


FIGURE 8. View of flame plating gun inside cubicle.

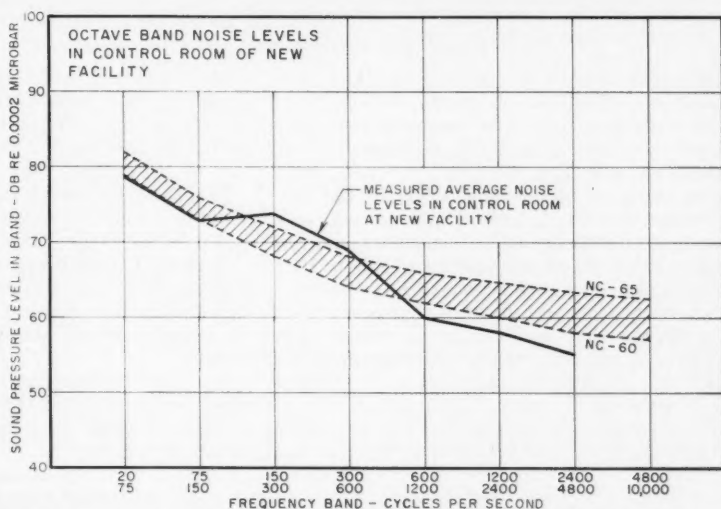


FIGURE 9. Comparison of noise levels in the control room with the NC 60-65 criteria.

detail shows the 2-inch "Tectum," or other similar sound absorptive interior treatment, applied to all wall and ceiling area, the glass fiber floated floor slab, and the 2-inch glass fiber treatment in the airspace.

The running weight per lineal foot for this inner wall construction is about 90 lbs, or about 10 times lower than the 900 lbs/lineal foot for the inner wall of the existing cubicles.

For the new cubicles the inner door was fabricated by Linde using the basic wall construction of 11 gauge steel panelling, and the outer door was a commercial sound retarding door. The window construction in each wall consisted of two panels of safety plate glass resiliently mounted with sound absorptive material between each pair of panes.

The conduit and service piping pass through an airspace between the two walls. Flexible rubber sections were installed in the piping, and loops were provided in the conduit. As a further precaution the penetrations in the inner wall were resiliently packed and sealed. A 2-in. thick glass fiber treatment was used on the exterior of the steel panel wall.

Each of the air handling ducts passing through the airspace to the roof was fitted with a commercial muffler, and in the airspace the duct or muffler was encased in concrete. Again the duct penetrations were resiliently packed and sealed.

In Figure 7 is shown a view of one of the

window sections with the sound absorbing treatment between the two 1/2-inch thick safety glass plates.

Finally, Figure 8 is a view of the interior of one of the new enclosures showing the Flame-Plating gun, the 2-inch "Tectum" interior surfaces, and the control window.

In Figure 9 are shown the octave band noise levels measured in the control room in comparison with the NC 60-65 criteria. These results show that over most of the frequency range the noise levels are below the selected noise control criteria, especially in the speech frequency range from 600 to 4800 cps. Thus, even with the marginal NR in the 3rd octave band very satisfactory noise control was achieved with a significant improvement over the previous facility. In a more critical installation, the NR in the 150 to 600 cps frequency range could possibly be improved by the use of 4 in. dense concrete instead of concrete block, without any change in wall weight, and by the use of more effective sound absorbing treatment in the airspace, or by the addition of sound absorbing treatment in the control room.

In conclusion, this enclosure design serves to illustrate how the use of space and sound absorptive materials, when combined with good design and installation of window, door and service connections, can compensate for limitations in wall weight.

CLEAN AIR CONFERENCE

THE UNIVERSITY OF NEW SOUTH WALES, Sydney, Australia, will hold a Clean Air Conference in that city on February 19 through 21, 1962. The first day will be devoted to general and administrative aspects of air pollution control and among the speakers will be Dr. W. F. Sheahan, Minister of Health, New South Wales; Dr. J. L. Sullivan, N. S. W. Dept. of Public Health; Mr. W. A. Damon, Dept. of Housing and Local Government, England; and Dr. M. Katz, Dept. National Health and Welfare, Ottawa, Canada. The remaining two days will be devoted to technical papers dealing with the evaluation, effects, and control of pollution. Approximately 30 papers are expected to be submitted for the technical sessions. The final program for the Conference will be circulated in December 1961 giving full information concerning contributors and the titles of papers to be presented. Complete preprints of papers will also be available to participants attending the technical sessions and will be distributed approximately one month before the Conference.

Applicants for registration should write to Dr. J. L. Sullivan, Conference Secretary, Department of Public Health, Box No. 31, George Street North Post Office, Sydney, Australia. Accommodations will be provided at Basser College, University of New South Wales, and reservations should be made as soon as possible.

An Inexpensive Multifrequency Pulse Generator for Calibrating Radiation Ratemeters

RICHARD P. MARQUISS* and RONALD L. KATHREN

Health Physics Branch, Mare Island Naval Shipyard, Vallejo, California

A pulse generator suitable for use in calibrating radiation rate-meters is described. Basically employing a simple multivibrator circuit, the device provides pulse rates ranging from 30 to 200 thousand pulses per minute. Stability and reproducibility of pulse rate was found to be good. The entire unit can be built from ordinary parts at a cost of approximately twenty-five dollars.

ONE of the most useful instruments employed by the health physicist is the radiation ratemeter. Ratemeter circuits are well known^{1, 2, 3} and vary in complexity from simple one-stage units up through very sophisticated multistage devices. Almost all are equipped with a means of adjustment in order to insure correct calibration.

Calibration checks are generally accomplished by one or more of the following methods:

- (1) Utilizing an external standard source of ionizing radiation.
- (2) Providing the ratemeter with an internal electronic calibration check circuit (line frequency).
- (3) Introducing an external pulse, similar to that of an ionizing event, directly into the ratemeter circuitry.

Each of these methods has certain advantages and disadvantages. However, most routine performance checks and servicing can be better accomplished with the aid of a pulse generator. One of the more desirable features of pulse generators is the ability to introduce a wide range of pulse frequencies into a circuit at a desired point. But despite this and other advantages, commercial pulse generators with variable repetition rates are generally priced so high as to preclude their purchase by a small health physics or industrial hygiene program.

Described below is a compact, easily constructed pulse generator (Figures 1, 2, and 3) of sufficient accuracy for everyday use which can be built for a total materials cost of less than twenty-five dollars. It produces a wide range of pulse frequencies and has provision for adjusting pulse shape and amplitude.

* Present address: U.S. Naval Radiological Defense Laboratory, San Francisco 24, California.

Theory of Operation^{4, 5, 6}

An essentially square wave is generated by a free running unsymmetrical cathode-coupled multivibrator. Frequency of oscillation is determined by suitable resistance-capacitance (r-c) networks selected by a multipole switch (SW₂). Several frequency ranges can thus be obtained. Variable frequency ranges are made possible by series connecting a potentiometer (R25) to any given r-c network.

The use of a multivibrator requires rigid voltage regulation.⁶ Ideally, it is necessary to use special electronic methods, but in this application adequate regulation is obtained through the use of two series connected gaseous regulator tubes (V₂, V₁). Adequate high voltage ripple filtering is accomplished by a standard r-c filter network.

Large voltage excursions, present at low frequencies in the multivibrator plate supply, tend to appear as part of the output signal unless adequately bypassed. A decoupling filter between the two sections of the pulse generator eliminates this difficulty.

The multivibrator cathode resistor (R7) is made variable in order to readily choose the best operating bias for the multivibrator and to compensate for tube aging or replacement. At certain settings, this potentiometer can cause the multivibrator to oscillate concurrently at two widely separated frequencies.

The essentially square wave from the multivibrator is coupled to a combination clipper-amplifier (V₂) by a simple r-c differentiation circuit which changes the wave form to alternating positive and negative spikes. An inverter-clipper (V₂A) acts to establish a nearly constant pulse amplitude for all repetition rates. Uni-

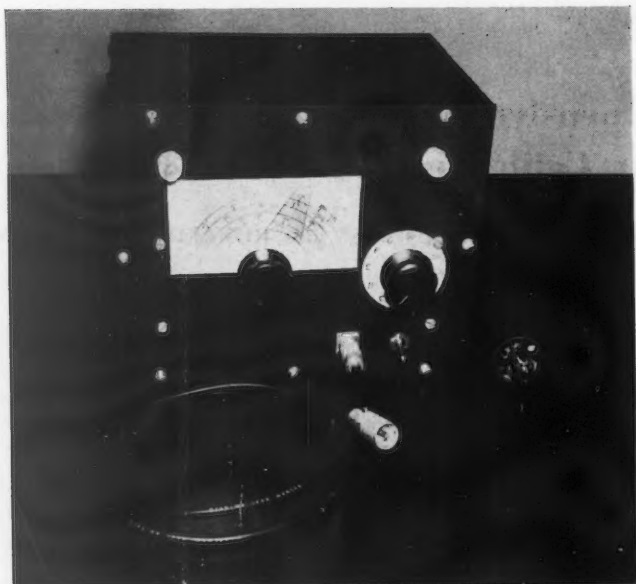


FIGURE 1. Front view of the assembled unit. Toggle switch controls power which is indicated by left neon bulb. Right neon bulb indicates pulses, and flashes may be counted as calibration check for low ranges. Right hand knob is range switch; variable ranges indicated by letters. Left hand knob controls potentiometer. Output is through RG-59/U coaxial cable. Note use of grounding house plug. (Official U. S. Navy Photograph.)

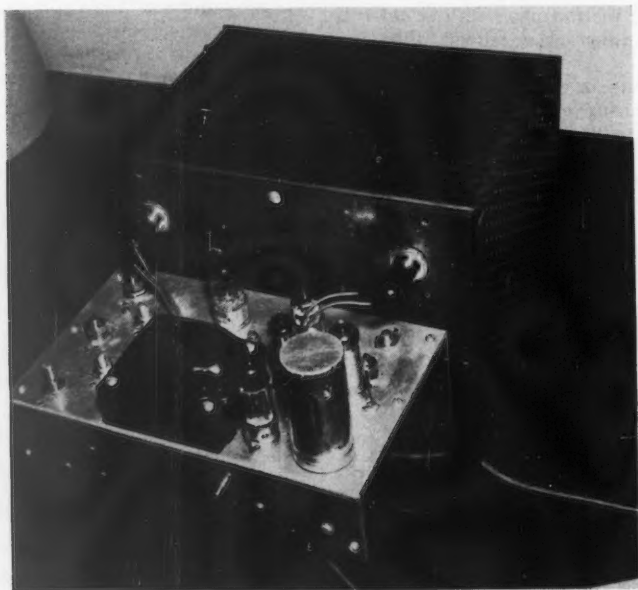


FIGURE 2. Interior view. Fixed range potentiometers shown at left of apron. Multivibrator cathode resistor (potentiometer) shown at center, above transformer. (Official U. S. Navy Photograph.)

clipper cathode resistor (R10) makes possible double pulses, unidirectional positive spikes and other common pulse shapes.

The output from the clipper-amplifier is coupled to a voltage dividing potentiometer (R12) through a relatively large capacitor which prevents further differentiation. The potentiometer provides the means of adjusting the pulse amplitude. It should be noted that both the capacitors in the output voltage divider circuit are high voltage types in order to withstand the highest detector voltage supply likely to be encountered.

Operation

The pulse generator described is provided with five fixed frequency ranges of 50, 500, 5K, 50K and 80K pulses per minute and seven overlapping variable ranges of from 30 to 220K pulses per minute. The pulse rates were chosen to provide calibration frequencies for the instruments in use at Mare Island Naval Shipyard. Different rates can be obtained by changing the values of the frequency determining r-c networks.

Adjustment and calibration were accomplished with an accurate automatic scaler-printer and a calibrated oscilloscope. Short term (one minute) warm-up drift was found to be less than three per cent. During continuous operation, drift was less than one per cent.

Reproducibility was also found to be good after relatively long periods (up to three months) of non-use. A change of less than one per cent was noted on most ranges, with the maximum change on any range being less than three per cent. These slight variations should not be great enough to interfere with normal

health physics usage. Annual calibration is recommended.

The pulse generator described has been utilized satisfactorily for the calibration of several types of ratemeters, including portable battery-powered survey meters. It has also been of value on the service bench to aid in locating problems in ratemeter circuits that are more complicated than those corrected by tube replacement.

Summary

A portable variable frequency pulse generator with variable pulse amplitude and shape is described. The unit can readily be used for calibration of radiation ratemeters, scalars and similar devices.

Stability and reproducibility of the pulse frequency and amplitude were found to be good. The entire unit can be readily constructed for a total materials cost of less than twenty-five dollars.

(Note: The views expressed herein are those of the authors and do not necessarily reflect those of the Department of the Navy or Mare Island Naval Shipyard.)

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Synthesis of Di-2-Naphthylthiocarbazone — A Simplified Method

DONALD M. HUBBARD

The Kettering Laboratory, Department of Preventive Medicine and Industrial Health, College of Medicine, University of Cincinnati, Cincinnati, Ohio

A simple, straightforward laboratory method is given for the preparation of di-2-naphthylthiocarbazone. The method requires no elaborate equipment and can be used for preparing this compound in a refined state, showing a molecular extinction coefficient of approximately 42,000 at wave length of 645 m μ . Since this compound is superior to dithizone when used for the determination of mercury in biological material (particularly urine) and since it is no longer commercially available, its preparation within the laboratory becomes a necessity. The starting compound, 2-naphthylhydrazine hydrochloride, is readily available.

DI-2-NAPHTHYLTHIOCARBAZONE is no longer commercially available for various reasons. Therefore it has become necessary again to prepare this valuable compound within the laboratory. Since the compound is so useful for the analysis of biological material (particularly of urine) for its content of mercury,^{1, 2, 3} a procedure for its easy and safe production in the laboratory appeared desirable. The method of synthesis published by Hubbard and Scott⁴ is laborious, difficult to manipulate, and employs as a starting material the potentially hazardous 2-naphthylamine.

Fortunately, 2-naphthylhydrazine hydrochloride is available commercially. The liberated free base can be treated with carbon disulfide to form an addition product. This addition product was described by Hauff⁵ as early as 1889, and later by Suprunovich.⁷ As noted by Hubbard,¹ when a saturated ether solution of this product was allowed to stand in a loosely stoppered Erlenmeyer flask for several days, a slow evolution of hydrogen sulfide resulted and di-2-naphthylthiocarbazide was obtained as a gray precipitate adhering to the sides and bottom of the flask. According to Preund the melting point is 137°–140°C., and by treatment of the compound with alcoholic potash, di-2-naphthylthiocarbazone can be formed.⁷ Finally, refinement of this product should produce a very pure grade of di-2-naphthylthiocarbazone with the molecular extinction coefficient of approximately 42,000 at 645 m μ .⁸

This is the basis of the method of synthesis to be described.

Experimental

Production of Free Base

Weigh out a 10-gram portion of 2-naphthylhydrazine hydrochloride. Dissolve it in one liter of hot distilled water. Add, with stirring, 350 ml of one per cent aqueous KOH solution and, using a Büchner porcelain funnel, filter upon a 7.0-cm No. 42 Whatman filter paper, and wash the precipitate sparingly with distilled water. Transfer the precipitate to a watch glass and air dry overnight. The final yield is approximately five grams of free base.

Formation of Addition Compound

Place 10 grams of the dry, free base in a large graduated Erlenmeyer flask and add one liter of diethyl ether. Stir with a Mag Mix electric mixing device, at setting 6, after inserting a plastic Teflon Magnetic Bar (3/4" × 5/16"). From a separatory funnel, add a mixture of 2 ml carbon disulfide and 20 ml diethyl ether, drop by drop. Stir for 30 minutes. Theoretically the mole ratio of free base to carbon disulfide is 2 to 1.



Formation of Di-2-naphthylthiocarbazide

Place the Erlenmeyer flask containing the addition compound in the hood, cover with an inverted 250-ml Griffin low form beaker, and allow to stand overnight. On the following day,

scrape down the sides of the flask with a long glass stirring rod having one end flattened and edged to permit easy removal of material clinging to the flask. Filter the precipitate onto a 7.0-cm No. 42 Whatman filter paper placed in a Büchner, porcelain funnel. Adjust the volume to one liter with fresh ether, and allow the Erlenmeyer flask to stand in the hood overnight. Approximately eight grams of material are filtered off, while the orange-colored, ether solution is saturated with the addition product.

The mother liquid is allowed to stand for 48 hours and the azide which forms and clings to the inside of the flask is scraped down and filtered off. It is then washed with fresh ether, air dried, weighed, and the melting point is determined. The orange-colored mother liquid is again saturated by the addition of four grams of the original eight grams of addition compound, and the flask is shaken, after which it is allowed to stand for 24 hours, and the undissolved material is again filtered off and combined with the remainder of the eight grams originally obtained.

Again the mother liquid is allowed to stand for 48 hours, after which the azide is removed, and melting point determined as before. The above process is repeated, possibly four times, until there is no evidence of further production of azide, and until the final compound remaining is the pure azide. The mother liquid is then discarded.

The di-2-naphthylthiocarbazine has a melting point of 138°–140°C. and its color is light gray.

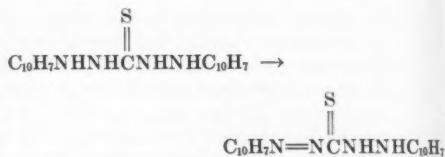


Formation of Di-2-naphthylthiocarbazine:

Twenty ml. of 50 per cent alcoholic potassium hydroxide is added to 0.5 gram of the dry azide in a 250-ml beaker, and mixed well with stirring rod, at room temperature. The mixture is neutralized immediately in a 2-liter beaker with one liter of dilute hydrochloric acid (40 ml of 6N HCl per liter), mixed well, and filtered on a 7.0-cm No. 42 Whatman filter paper. The precipitate is washed well with distilled water and air dried overnight.

The dry, but impure, di-2-naphthylthiocarbazine obtained by treating two 0.5-gram portions of the azide, is dissolved in 50 ml of reconditioned chloroform⁸ at room temperature, and the chloroform solution is filtered through a

Büchner fritted pyrex funnel (60-ml capacity and porosity M). The funnel is washed well with approximately 50 ml of additional chloroform. The chloroform filtrates are combined and placed in a Petri dish of 150-mm diameter. The excess of chloroform is then evaporated off at room temperature. By means of a spatula, the di-2-naphthylthiocarbazine is removed from the Petri dish. This material compares favorably in purity with that which was obtainable commercially.



The end product is then refined by the procedure described in an earlier publication.⁸

Discussion

Ten grams of pure 2-naphthylhydrazine will produce approximately two grams of di-2-naphthylthiocarbazine, which, by further purification, gives 1.2 grams of the purified material. This yield is 11 per cent of theory. The yield is slightly lower than that of the method previously published, but it may be susceptible of increase through further experimentation.

The main impurities encountered are those formed when di-2-naphthylthiocarbazine is treated with alcoholic potash solution. Upon neutralization, potassium chloride is formed, which is almost completely removed when the chloroform solution of the dry material is filtered. During the mutual oxidation-reduction which takes place, 2-naphthylthiosemicarbazide and 2-naphthylamine are formed as by-products. The latter by-product is water soluble and is removed by washing. However the first by-product is not removed until the final step of purification, when it is removed by the alcohol wash.⁸

Laboratories interested in using di-2-naphthylthiocarbazine in the refined state will be able to use this method of synthesis, since it is simple, straight-forward, and requires no elaborate equipment. The final amount of refined product, although only 1.2 gram, is an ample supply for many determinations of mercury.

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CALIBRATION OF MICROPHONES

THE NATIONAL BUREAU OF STANDARDS maintains a service program for the calibration of microphones for use as secondary standards by governmental or private organizations. Those interested in audiometry or the sound measurements in industry may well find this service of great value in assuring that the microphones used in their work are functioning properly and giving measurements of sufficient accuracy. The measurement of high sound pressure levels should be made with microphones which have been calibrated at high intensity sound levels. Secondary standard microphones, which are used to calibrate other microphones and sound-measuring instruments, are calibrated against a set of primary standards in the Sound Laboratory of the National Bureau of Standards. Current research at the Bureau is aimed at extension of the range of microphone calibration to infrasonic and ultrasonic frequencies, the extension of the amplitude range to higher pressure sound levels, and increased accuracy and precision of operation. In the near future microphone calibration facilities will be expanded when the Bureau moves to its new site in Gaithersburg, Maryland. The new facilities, including an enlarged double-wall anechoic chamber, will permit expansion of the NBS research program to meet the increased demands of industry, science, and national defense.

The Bureau performs two types of calibrations known as the pressure calibration and the free-field calibration. In the pressure calibration the secondary standard is compared to the primary standard microphone of the NBS in the same chamber in which the primary calibrations are made. Calibrations are performed in the coupler at discrete frequencies from 50 cycles per second (cps) to 10 kilocycles per second (kc/sec). Secondary free-field calibrations are performed in an anechoic chamber for many types of microphones over a frequency range of 50 cps to 15 kc/sec. Microphones are calibrated at normal or grazing incidence by comparison with the NBS primary standard condenser microphones. With type-L laboratory microphones calibration in the anechoic chamber is more costly and not as accurate as performing a pressure calibration and using a standard diffraction correction to the pressure response at each frequency.

This calibration service is one of a series of such services performed by our National Bureau of Standards to insure the accuracy and precision of necessary scientific measurements in both research and field application situations.

Air Flow Requirements for Underground Parking Garages

GEORGE M. HAMA, M.S., WILLIAM G. FREDRICK, Sc.D., and
HARRY G. MONTEITH, M.S.

Bureau of Industrial Hygiene, Detroit Department of Health, Detroit, Michigan

Extensive studies correlating carbon monoxide concentrations with traffic density, car capacity and ventilation rates in a two and three-level underground parking garage showed that 500 cubic feet per minute of ventilation must be provided for each parking space in the garage to maintain hygienic standards based upon carbon monoxide and customer acceptance based upon smoke and odor. Provisions should be made to reduce ventilation rates to 350 cubic feet per minute per space during normal load periods. Hygienic standards for peak load operations, fleet type garages and general service type garages are discussed.

Introduction

THIS paper seeks to answer the following questions:

- (a) On what basis should the ventilation rates be determined for an underground garage? Air changes per hour? cfm per sq. ft.? Number of cars? Other methods?
- (b) What carbon monoxide concentrations should be allowed? Maximum? Average?
- (c) What ventilation rate in cubic feet per minute will give adequate control of carbon monoxide?
- (d) What fluctuations of carbon monoxide levels can be expected in actual operation? Should flexibility of ventilation rate be planned for by providing a number of fans, possibly with two-speed motors?

Ventilation Rate in Existing Garages

There have been a number of municipal public underground parking garages built since the first garage^{1, 2} of this type went into operation nearly twenty years ago. Table I compares six larger garages which have been in operation for a number of years.^{1, 2, 3, 4, 5, 11} It can be seen from the tabular information that the ventilation rates vary widely (over 200%) regardless of the basis on which they are compared.

The question raised in comparing such data is what ventilation rate will be satisfactory. Are some of the garages over-ventilated or are the other garages under-ventilated?

Basis for Determining Ventilation Rate

There have been several methods used in setting up ventilation rates for underground parking garages, these are:

- (a) Specifying ventilation rates in air changes per hour.
- (b) Specifying ventilation rates in cubic feet of air per minute per sq. ft. of floor area.
- (c) Specifying ventilation rates in cubic feet per minute per car in operation or on the basis of car capacity of the garage.

Air Changes per Hour (ACH)

This is an unsatisfactory method, first because it bases the ventilation rate on a volume of space and not on dilution of the contaminant. Second, if the volume of space is large (which allows for greater dilution), the cfm ventilation rate will be larger than for a smaller volume space where the dilution is less. Air changes per hour, therefore, penalize a large volume space, and comparatively do not give a high enough ventilation rate in a small space.

Several building codes,^{6, 12} have suggested a ventilation rate of six air changes per hour for underground garages. In the authors' knowledge this is not based on carbon monoxide dilution but is more probably associated with the potential fire hazard from the gasoline leaks and vapors escaping from automobiles.

Volume of space varies in the several underground garages considered in Table I. The ceiling clearance height alone varies from seven feet to nine feet six inches. It appears that setting a rate based on air changes per hour will not be correct for all garages with varying volumes and car capacities.

Cubic Feet of Air per Minute per Square Foot of Floor Area (cfm/sq.ft.)

This method *per se* is not a sound basis for determining air flow rates for carbon monoxide

control from motor vehicles because it does not base the air flow rate on the contaminant given off, but merely on a building dimension, the floor area. However, it is a more logical basis than the air change method because the floor area is related to the number of cars a garage will hold. However, space requirements per car, aisle width and ramp area, may vary considerably with garage design. Floor area per car based on six garages in Table I varies from 208 to 388 sq. ft.

Ventilation rates which break down the floor area into two different sets of areas, car storage or parking area, and ramp area should give closer approximation of the ventilation rate required. Some suggested rates^{6,7} have been 0.5 to 1.0 cfm per sq. ft. of car storage, 2.0 cfm per sq. ft. of ramp area, and 3.0 cfm per sq. ft. of entrance drives.

Cubic Feet per Minute per Car in Operation

This method appears to be the most logical basis for determining the rate of ventilation. The quantity of contamination such as carbon monoxide, oil vapor, unburned hydrocarbons, oxides of nitrogen and other contaminants varies directly with the number of cars in operation and not with floor area, or volume of space.

The figure of 5000 to 6000 cfm per car in operation has been used as a basis for design of garage ventilation.⁷ It is probable that this figure is not too accurate as it was based on some investigations made over 25 years ago on a car idling. In underground parking garages cars (in operation) will be idling, accelerating, decelerating, starting with full choke, and running under heavy load conditions going up the ramps. Also, while the efficiency of the auto engine has increased in the last 25 years, the horsepower has also greatly increased.

In order to determine the air volume based on the number of cars, however, it is necessary to know in a garage of given capacity how many cars can be expected to be in operation at one time during the various periods of the day, how long the engines will be running and how much air flow will be required to dilute the contaminant from a car to a satisfactory level. It was the object of this study to ascertain these factors.

The purpose of the ventilation is to reduce or control the concentration of hazardous and nuisance gases to a satisfactory level and to provide acceptable odor and visibility conditions.

Among the contaminants present in the exhaust gases are carbon monoxide, carbon dioxide, oxides of nitrogen, aldehydes, phenols, oil vapor, unburned hydrocarbons and carbon particles.

TABLE I
Comparison of Ventilation Rate in Several
Underground Parking Garages

Name of Garage and Location	Levels	Total* Capacity No. cars	Total Ventilation cfm	cfm per car	air** change per hour	cfm per sq. ft.
Union Square San Francisco, Cal.	4	1700	390,000	230	6.6	0.9
Pershing Square Los Angeles, Cal.	3	2000	488,000	244	6.3	0.9
Mellon Square Pittsburgh, Pa.	6	897	245,340	273	7.8	1.0
Grant Park Chicago, Ill.	2	2359	1,766,400	750	16.8	2.2
Grand Circus Park Detroit, Mich.	2-3	1100	540,000	490	11.3	1.5
Military Park Newark, N. J.	3	1031	400,000	388	10.0	1.15

* Floor area per car ranges from 208 to 388 sq. ft.

** Ceiling height of various levels varied from 7'-0" to 9'-6"

Although all these contaminants contribute to the unsatisfactory air conditions, carbon monoxide is one of the most important factors healthwise and also has been found to be a good index of haze caused by oil vapor and particulate matter. It is easy to measure by means of recording instruments. Carbon monoxide level was therefore used as an index of satisfactory ventilation.

Permissible Levels of Exhaust Gas

Fleet Type Garages

The permissible levels of carbon monoxide should be a maximum concentration of 200 ppm for a period not exceeding one hour and an average concentration not exceeding 50 ppm during any 8-hour operating period. A fleet type garage is one in which the garage is quickly loaded once and then unloaded once in any 8-hour period with little or no vehicular activity in the interim.

General Service Type Garages

The permissible levels of carbon monoxide should be a maximum concentration of 100 ppm and an average concentration not exceeding 50 ppm during any 8-hour operating period. A general service type garage is one in which a fairly even level of activity is maintained over any 8 hour period.

Comments. It is quite possible that a single structure might be used as a general service type garage for one 8-hour period during the day and as a fleet type for another 8-hour period in the evening because of theatrical or special event parking. It should be noted that customer acceptance declines rapidly because of objectionable odor, smoke and eye irritating conditions at about the 100 ppm carbon monoxide concentration level.

Basis for Permissible Levels. Although nearly all of the individual components of automobile exhaust gas are detrimental to health if sufficient quantities are present in the atmosphere breathed by man, the predominance of the asphyxiant gas carbon monoxide in the mixture causes its concentration to be the governing index of relative hazard for acute exposure. At physiologically acceptable concentrations of carbon monoxide in respirable air, the quantities of the other components of exhaust gas are too low to produce acute health effects of import. Little is now known concerning the long term effects on health of repeated day in, day out breathing of air contaminated with automobile exhaust gas up to present acceptable limits of carbon monoxide exposure. Therefore, all ventilation criteria herein established are based on carbon monoxide concentrations, and to a lesser extent on odor and visibility acceptability.

Concentrations of carbon monoxide in respirable air which will not impair coordination, judgment, reaction time, visual acuity or well being are dependent upon a complexity of factors among which are duration of exposure, barometric pressure, activity, age, sex, lung efficiency, and the coexistence of other diseases or conditions which are likely to cause cellular anoxia. For example, a healthy male adult at sea level and at rest can breathe air containing 200 ppm of carbon monoxide for about two hours, at moderate activity for about one hour and at heavy labor for about 45 minutes without significantly demonstrable functional or health impairment.⁸ Similarly, he could tolerate 100 ppm of carbon monoxide for about eight hours at rest, four hours at moderate activity and three hours at heavy labor. Similar tolerance times for women, small children and people with certain disease would be less than these. Most well people at barometric pressures near those of sea level can tolerate 50 ppm and all of the population can tolerate 30 ppm indefinitely.²⁰

Method of Approach

To determine air flow requirements for underground parking garages it was decided to

study an underground parking garage during actual operation. The garage selected was the Grand Circus Park Garage, Detroit, Michigan.

This structure consists of a two-level, pie-shaped (quarter circle) underground garage west of the main street (Woodward Avenue) in the downtown area and a three-level, pie-shaped (quarter circle) underground garage east of the main street. The two sections of the garage are connected by a tunnel below the main street. The distribution of the air flow in the garage is good. Air is supplied mechanically to the garage through a large supply opening at the point of the pie-shaped sector. A series of exhaust openings around the periphery or circumference of the pie-shaped sector removes the air into an outer plenum where its flow is effected by a centrifugal fan. From here it is discharged to the outdoors.

The exhaust ventilation is provided by four centrifugal fans with two-speed motors. Two are on the tri-level east section and one on the bi-level west section and one in the connecting tunnel.

To determine average carbon monoxide levels a Lira MSA infrared recording carbon monoxide indicator was placed in the exhaust fan plenums of the east and west garage. This instrument was calibrated from time to time as required. The recorder was provided with chart paper with line divisions on the abscissa representing 15 minute intervals, line divisions on the ordinate representing zero to 100 ppm carbon monoxide, the smallest division representing one ppm. The instrument was highly sensitive and could be easily read to two parts per million of carbon monoxide by volume.

To ascertain the number of cars in operation during the carbon monoxide sampling periods, customer tickets stamped to the nearest minute were obtained from the management. Each ticket represented a car coming into the garage and leaving the garage. The time of day to the minute the car came in and went out of the garage was stamped on the ticket by the attendant. The level at which the car was stored was indicated by the color of the ticket. Over 5,000 tickets were tabulated.

To determine the average duration time of operation of the cars in the garage, a time study was made of the time of motor operation of cars entering and leaving the garage. This was accomplished by the use of observers with two-way radio telephones located at the entrance, exit and within the garage. By means of this type of communication a car entering or leaving could be identified and precisely timed to the nearest 0.1 minute. Over 300 cars were timed at

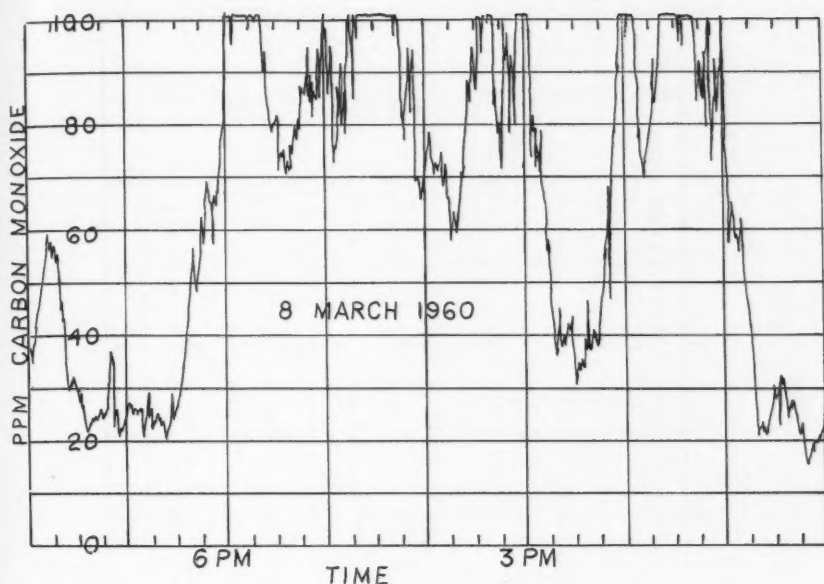


FIGURE 1.

various times and days. To determine distribution and time lag for the contaminant gas to pass in any area in the garage into the exhaust grill and through the plenum to the sampling hose of the recorder, carbon monoxide was released in the garage at various locations with no cars present, and the time required for the carbon monoxide to reach the recorder and register on the chart was noted.

Discussion of Data

Average Carbon Monoxide Concentrations

Figure 1 indicates a typical chart reading obtained from the infrared carbon monoxide recorder. Daily recordings were made and tabulated for a 4-month period. The vertical lines represented time intervals of 15 minutes. The chart moves from right to left. The average concentration can be estimated within 2 ppm at any 15 minute interval of garage operation and could be identified with the time of day by the chart clock numbers. By making an integrated average from totaling 15 minute time interval concentrations and dividing by the number of 15 minute intervals in an operational day period an average day's exposure could be calculated. The average exposures in the Detroit Garage with the fan running at 100% of full capacity and 50% of total capacity are as follows:

Fan on high speed (full capacity)	31 ± 12 ppm
average integrated carbon monoxide concentration	
Fan on low speed (half capacity)	58 ± 26 ppm
average integrated carbon monoxide concentration	

Relationship between CO Concentration, Number of Cars Running, and cfm. By ascertaining the total number of cars running per hour from customer ticket stubs (with incoming and leaving times punched on them) and noting the carbon monoxide meter charts, a relationship between carbon monoxide concentration and cars in operation could be determined. Graphs (Figures 2 and 3) were plotted to show the relationship. In these graphs the number of cars running during each hour was plotted against average hourly carbon monoxide concentration. The graphs are plotted with the data from conditions of both high and low speed fan operation. These graphs indicate that the curve is a straight line relationship, the slope of which depends on the ventilation rate. From the slope and intercept of the curves thus obtained, an equation with three variables: Carbon monoxide concentration, numbers of cars running per hour, and ventilation rate in cfm can be set up.

The equation is:

$$C = KN/Q$$

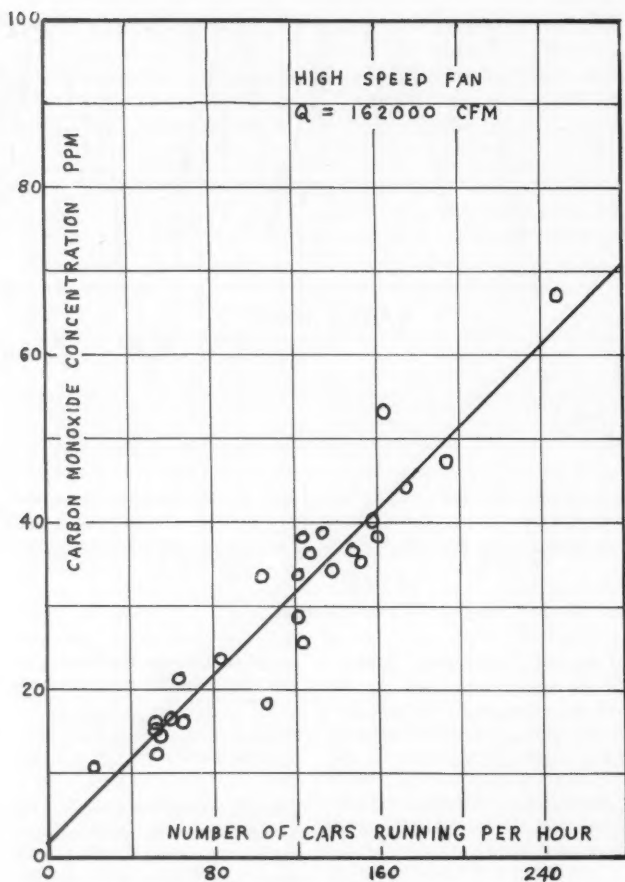


FIGURE 2.

where

C = concentration of carbon monoxide in ppm,

N = number of cars running per hour,

Q = ventilation rate in cfm exhausted,

K = constant (empirical).

This constant from the high speed fan data is $K = 42500$, and from the low speed fan data the constant is $K = 41300$. Average $K = 41900 \pm 600$.

Time of Operation of Each Car Running. A time study of period of operation of each car coming in or leaving the garage indicated a range of times of operation from 0.5 to 5.5 minutes. The average times of operation for the two level west section are as follows:

Upper level incoming	1.64 minutes
Upper level outgoing	1.62
Lower level incoming	1.94
Lower level outgoing	2.11
Over all average	1.8 ± 0.2

Volume of Air Required (cfm) to Obtain a Required Carbon Monoxide Concentration in a Garage with any Number of Cars Operating at One Time

From the data giving number of cars operating hourly, the number of cars operating simultaneously can be calculated, if we know the average time interval of car operation. This has been determined to be 1.8 ± 0.2 minutes. (Number cars running at any instant) = $(1.8/60) \times$

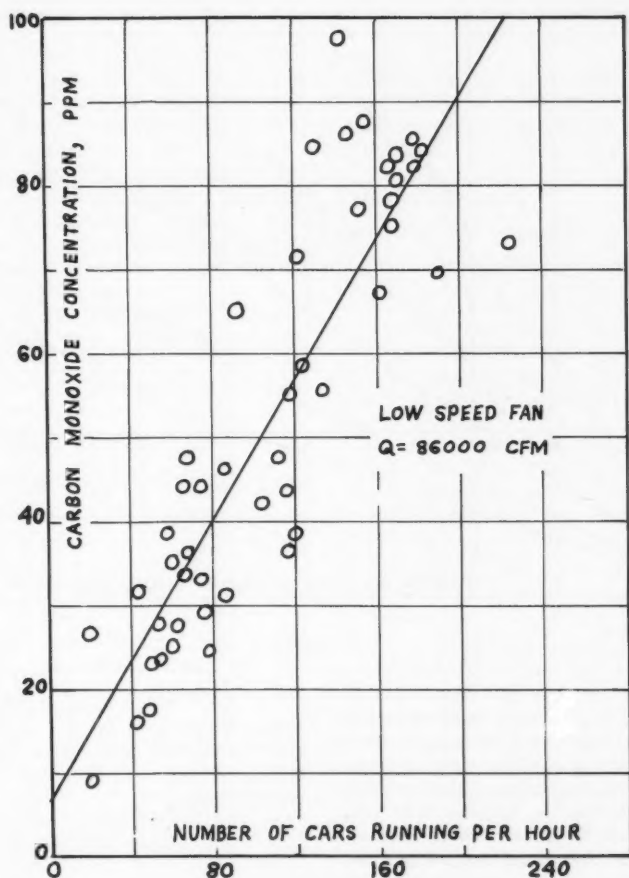


FIGURE 3.

(number of cars running per hour.) From this and the previous equation ($C = KN/Q$) the following equation is derived which can be used to determine the required exhaust air flow (Q), to control the exhaust gases to any carbon monoxide level (C) with any number of cars running at one instant (n).

The equation is:

$$Q = K'n/C,$$

where

$K' = 1.38 \times 10^6 \pm 20\%$ (a constant),

n = number of cars running at one instant,

C = carbon monoxide concentration in ppm.

Figure 4 graphically shows the relationship between cars running simultaneously, ventilation rate and carbon monoxide levels. The graph

gives air flow requirements in millions of cubic feet of air per minute, with various number of cars in operation at any one instant for carbon monoxide concentrations of 50, 75, 100, 150, 200 parts of carbon monoxide per million parts of air by volume. If the number of cars to be run at one time can be determined, the design air flow can be ascertained for the carbon monoxide exposure level desired.

Air Volume Necessary to Dilute Carbon Monoxide from One Car to 50 and 100 ppm. It is also of interest to determine the amount of air in cfm necessary to dilute the carbon monoxide from an average car in operation in the garage to 50 and 100 ppm of carbon monoxide, respectively. The graph of Figure 4 and the equation indicates that 27600 cfm is required to dilute the carbon monoxide from one car to 50

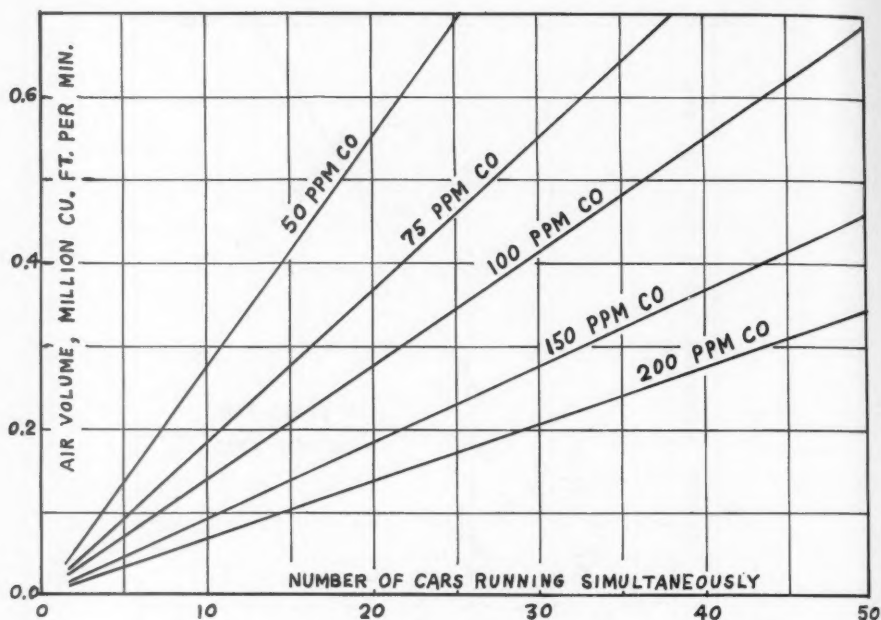


FIGURE 4.

ppm, and 13800 cfm is required to dilute the carbon monoxide from one car to 100 ppm.

The average car in this case is a composite ranging in size from a small horsepower foreign car such as the Volkswagen to a high horsepower American car such as the Cadillac. The composite operation of the car includes accelerating, decelerating, high load, road load, idling and starting operations. To determine this by a direct method, a great many different types of cars would need to be studied individually and a weighted average determined.

Estimate of Number of Cars in Operation at One Time Based on Total Capacity of Garage. If designing engineers were able to ascertain the average number of cars that would run simultaneously based on the total car capacity of the garage, and also the number operating simultaneously during peak periods, they could accurately determine the air flow requirements for garages of this type for control to an average exposure level of 50 ppm carbon monoxide and 100 ppm carbon monoxide for short peak loads.

Accurate counts of the number of cars in operation in the garage indicate that the average percentage of cars running based on total capacity is $1.18 \pm 0.05\%$ of design capacity. The car counts also indicate that maximum number of cars run at once during short peak periods is $3.2 \pm 0.3\%$ of design capacity.

Design Data Based on Capacity of Garage

The design data for 2-level garages of this type (of equally good air distribution, similar volume to car ratio and similar ramp distances) are as follows:

Air Volume to Control to an Average Level of 50 Parts Carbon Monoxide per Million Parts of Air.

Air flow in cfm required to control to 50 ppm carbon monoxide =
 $27600 \times (\text{total capacity in number of cars}) \times 0.0118$

Solving for known constants, the equation can be simplified to:

Air flow, $\text{cfm}_{50} = (\text{total capacity in number of cars}) \times (350 \pm 70)$.

Air Volume to Control to Maximum Peak Loads of 100 Parts Carbon Monoxide per Million Parts of Air.

Air flow in cfm required to control to 100 ppm carbon monoxide = $13800 \times (\text{total capacity in number of cars}) \times 0.035$.

Solving for known constants, the equation can be simplified to:

Air flow, $\text{cfm}_{100} = (\text{total capacity in number of cars}) \times (500 \pm 100)$.

Requirements for Multi-Level Underground Parking Garages Other Than The Two Level Garages. Garages having more than two levels will obviously have a slightly increased time interval of operation of the car motor. Data from the garage studies indicates an average increase in time of motor operation of 0.2 minutes per level. This will increase slightly the concentration of carbon monoxide given off per car. The following calculated design figures are recommended for over all air flow figures for parking garages of various levels.

Ventilation rates required to control carbon monoxide concentration to an average level of 50 ppm and a peak level of 100 ppm based on total design capacity of garage (in number of cars) are as follows:

CFM REQUIRED

Level	(total car capacity of garage)	CFM
1	"	$\times 450 \pm 90$
2	"	$\times 500 \pm 100$
3	"	$\times 550 \pm 110$
4	"	$\times 610 \pm 120$
5	"	$\times 670 \pm 140$
6	"	$\times 720 \pm 150$
7	"	$\times 780 \pm 160$
8	"	$\times 840 \pm 170$

The total air flow should be distributed in approximately equal quantities on each level.

Distribution and Sources of Air

The use of ramps as a fresh air supply in underground parking garages is not a good practice, as street level air carrying considerable carbon monoxide will not produce as much dilution per cfm as less contaminated air. A location of a clean air source away from the street should be considered. Carbon monoxide levels in downtown Detroit at the street range from 0 to 100 ppm with a median of 9 ppm.¹³ The readings were made with an infra-red recording carbon monoxide meter during a 21-week period.

The location of air inlets and outlets should be made so that equal quantities of air flow sweep the entire garage space. An example of excellent design of a rectangular garage is the Grant Park Garage in Chicago. Here 27 axial flow fans exhaust air from the garage into discharge wells opening to the surface. The fans are equally spaced along one long side of a rectangle. The opposite side provides large area air inlet gratings covering nearly the entire portion of the opposite wall. The air comes in from bench openings at the park on the surface away from the street.

Ticket booths and attendant booths should be supplied with heated outdoor air in sufficient quantity to effect an outflow of air through the open booth window of at least 50 linear ft/min.

No exhaust ventilation should be used in such booths.

Offices, waiting rooms, and other occupied spaces should be pressurized with an excess of heated outdoor air to prevent infiltration from the garage.

Fluctuation of Activity

A fifteen hour period from 9:00 a.m. to 12 M appears to be an interval of high activity. Periods from 12 M to 9:00 a.m. are marked by a great reduction in activity. To reduce power costs and heating load (if the garage is heated), therefore, it is recommended that several fans with two speed motors be provided so that in periods of relative quiescence the ventilation rate can be reduced.

Manual fan operation has been found not to be dependable. The operations of the fans when a proper schedule has been determined should be made by means of time operated switches or switches operated by carbon monoxide monitoring equipment.

Summary

The design air flow rates for existing underground parking garages vary considerably.

Most designs are based on rates given in air changes per hour or cfm per sq. ft. A more sound basis is cfm per car in operation.

Recommended carbon monoxide levels are given for fleet type and for general service garages.

Carbon monoxide levels measured are correlated with air flow data, number of cars in operation, average time of operation to give design data based on the car capacity of the garage.

Acknowledgments

Grateful acknowledgment is made to Messrs. K. J. Bonkowski, R. O. McLean and L. C. Curley of the Bureau of Industrial Hygiene, Detroit Department of Health, who assisted in the field work and instrumentation for the Parking Garage Study.

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AIR POLLUTION MONITORING

THE ESTABLISHMENT OF A CONTINUOUS AIR-MONITORING PROGRAM to provide vital information on air pollution levels in American cities has been announced by Surgeon General Luther L. Terry of the Public Health Service. Designed to provide automatic measurement and analysis of the continuously fluctuating levels of pollutants in urban air, the program is based on special air-monitoring equipment located in the center of the city area of eight major U. S. communities—Chicago, Cincinnati, Detroit, Los Angeles, New Orleans, Philadelphia, San Francisco, and Washington, D. C.

Built and equipped by the Public Health Service at a total cost of \$300,000, the new equipment went into operation this fall and provides, at the outset, automatic and continuous sampling and analysis of seven gaseous pollutants — sulfur dioxide, nitric oxide, nitrogen dioxide, carbon monoxide, ozone, total hydrocarbons, and total oxidants. In addition to automatic gas sampler-analyzers, the stations will also house supplementary equipment to provide information on particulate pollutant concentrations, pollutants washed out of the atmosphere by rainfall, and measurements of wind turbulence at the sampling sites.

Continuous air monitoring is not intended to supplant the periodic sampling conducted by the National Air Sampling Network of the Public Health Service, which has been in operation since 1955. The National Air Sampling Network provides information, at more than 250 urban and rural sites, on average levels of particulate matter and of radiation; and, at 50 sites, on the average levels of two gaseous contaminants — sulfur dioxide and oxides of nitrogen.

The new continuous-monitoring stations will be operated by employees of the participating cities, all of whom have received special training at the Public Health Service's Robert A. Taft Sanitary Engineering Center in Cincinnati, Ohio. The first of the eight cities to begin operations was Cincinnati, which dedicated its station on October 23 to mark the beginning of Cleaner Air Week. The other stations are expected to have begun operations by the time this item goes to press.

Management of Airborne Wastes in a Nuclear Economy—The Role of Meteorology

DONALD H. PACK

Environmental Meteorological Research Projects, Meteorological Research Projects Branch, U.S. Weather Bureau, Washington, D.C.

The increasing utilization of nuclear energy will create new types of airborne waste problems. Creation of a "neighborhood" of multiple reactor sites (relatively) close together will require airborne budget calculations. In anticipation of these requirements meteorologists are extending their research into larger scales. Some of the new techniques for observation and calculation are presented. Included are radar tracking of tracers to distances of 100 miles, new fluorescent dye experiments, and new wind measuring techniques.

THIS article refers to a "nuclear economy", which might seem to make the remarks applicable only at some future date. However, I believe that the United States already has such an economy. Figure 1¹ shows the distribution of reactors now built, building, or planned in the United States. The numbers in the circles represent the number of reactors at one general geographical location. There are a total of 181 reactors illustrated in this diagram. This assembly of the "big picture" may come as a surprise, even for someone familiar with the nuclear field.

The usual concept of a nuclear economy occasions a mental image of an entire nation's electrical grid, powered by a multitude of reactors, the oceans traversed by fleets of nuclear ships, and radioisotopes in every industrial plant. The transition to such a situation would be so gradual we would probably never notice it, and it appears one can discuss the problem of airborne waste in the context that a nuclear economy now exists even though it may presently be smaller than other technologies. This point needs to be constantly kept in mind, lest the real and significant problems of nuclear waste disposal are set aside for tomorrow's thought and research. In the field of meteorology, research performed 30 years ago supplies the workaday tools for today's applications. The problem has been sharply focused by Hydeman and Berman,² and the following is a partial quotation from their study of nuclear maritime activities.

"...as peaceful atomic energy activities of all types increase in number and size, the radiation health and safety problems will unquestionably become more complicated... clearly the time is at hand when we must begin to give thoughtful and imaginative consideration to these needs."

The total problem of waste management is very complex, and involves many fields and specialties, including, for example, medicine, reactor physics, economics, meteorology, public relations, etc. The following brief discussion is deliberately limited to a look at meteorological studies, but with the recognition that many factors must be considered in the total problem.

The meteorologist's role in the control of airborne waste is almost always that of a consultant. Atmospheric dilution cannot in any way diminish the amount of material that is released to the atmosphere, it simply reduces the concentration of such material. "Management" of airborne waste is intended to mean active, rather than passive, supervision over any airborne radioactive material emitted, and management is used in the broad sense to mean not only the control of effluent volumes, but also quantitative and continuing knowledge of "what" has gone "where", "when", including an idea of the rate of, and total accumulation of, environmental radiological exposures.

Meteorology can be used in a number of ways in airborne waste management. It can be used in the selection of a facility location, and the arrangement of reactors, effluent stacks, etc. on an individual site. It can be used climatologically during design to determine the needed capacity of waste disposal facilities. Meteorology can be used in operations, either to turn the procedure on and off, or to accelerate or decelerate waste disposal, depending upon the capacity of the atmosphere to dilute the material. The amount and type of meteorological effort required will differ greatly from site to site and most installations will need only a limited amount of relatively uncomplicated measurements. Other installations may require quite elaborate en-

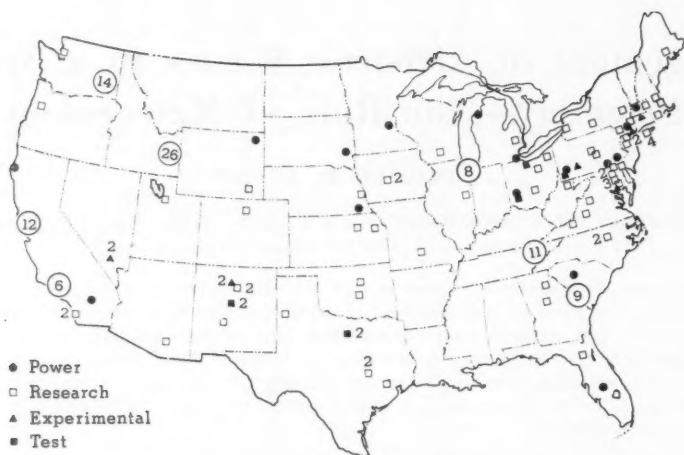


FIGURE 1. U.S. Nuclear Reactors (existing, under construction, proposed).

vironmental programs or may decide to contribute to the "state of the art" by supporting meteorological research.

With this proviso in mind it can be seen (Figure 1) that the problem of the creation of a neighborhood of multiple reactor sites is already with us, since there are installations where ten or more reactors exist within a few miles of each other. Quantitative evaluation of the airborne waste from such sites require radiological budget calculations. Such calculations are complex and time consuming, both because the nature of the sources may be complex and because the atmosphere behaves in various and diverse ways from day to day. What then can meteorology do to make this work practical?

In anticipation of such problems, recent research and development, supported by the Atomic Energy Commission and by the Public Health Service, have developed a number of promising tools and techniques which can improve our knowledge of the dispersion of radionuclides. These tools include both theoretical and instrumental techniques.

One of the most useful of recent developments is the use of wind direction fluctuations statistics^{3,4} to predict atmospheric diffusion. In the past most computations of airborne concentrations have been based on the work done in the early 1930's by Sir Graham Sutton in England. His prediction equation with its three coefficients, C_y , C_z , and n , has proved extremely useful, but the relationship of the coefficients to wind measurements is difficult to determine. On the other hand, we can watch a vane swing in the wind and intuitively feel that the motion of

material in the atmosphere should in some way be related to these fluctuations. Figure 2 is a generalized diffusion equation with only the assumptions of a Gaussian distribution of concentration about a plume center line and mass continuity. The important parameters for this discussion are the standard deviation of the plume dimensions in the lateral and the vertical directions. Recent work has shown that the fluctuations of the wind as measured by a wind vane can be very directly related to the plume

GENERALIZED DIFFUSION EQUATION

$$X = \frac{Q}{2\pi\sigma_y\sigma_z\bar{u}} \exp\left[-1/2\left(\frac{y^2}{\sigma_y^2} + \frac{z^2}{\sigma_z^2}\right)\right]$$

X = CONCENTRATION.....units/m³

Q = SOURCEunits/sec

y = CROSSWIND DISTANCE..... meters

z = VERTICAL DISTANCE..... meters

\bar{u} = MEAN WIND SPEED m/sec

σ_y = STANDARD DEVIATION OF PLUME WIDTH

σ_z = STANDARD DEVIATION OF PLUME HEIGHT

RELATION TO SUTTON

$$\sigma_y^2 = 1/2 C_y^2 x^{2-n}$$

$$\sigma_z^2 = 1/2 C_z^2 x^{2-n}$$

FIGURE 2. Generalized diffusion equation.

dimensions, σ_y and σ_z , and through them to atmospheric concentrations.

The data acquisition from the wind vane is quite straightforward. A suitably sensitive instrument is recored on a conventional wind direction recorder and the angular values of the wind fluctuations are extracted at short intervals to enable the calculation of the running mean standard deviation of the angular fluctuations. In practice this usually means speeding up the chart drives to enable the reading of data at intervals of five seconds or less. The most important portion of the analysis is the averaging technique used. While the reasoning behind the technique is quite sophisticated (see Hay and Pasquill⁴), the result is one of great simplicity, which says in essence that the standard deviation of a plume of diffusing material, σ_ϕ , is described by the running mean standard deviation of a wind vane, σ_ϕ , averaged over an interval (s). $s = T/\beta$, where T is the time for the plume to travel from its origin to the point of sampling, β is a "conversion" factor from the Eulerian (fixed point) wind vane measurements to the Lagrangian (moving coordinates) plume dispersion. Numerous experiments indicate $\beta \sim 4$. As an example, let us assume we have a wind instrument at the point of plume origin, and a sampler at 1,000 meters down-

wind. The average wind speed is, say, 5 meters per second. Then $T = 200$, $T/\beta \sim 50$ seconds. Therefore, the angular wind trace fluctuations are first averaged over a 50 second interval, and then the running mean standard deviation computed from these average values.

There are certain restrictions in the use of this technique, which requires reasonably steady weather conditions, sufficient data to insure statistical reliability, etc., but these qualifications are equally applicable to previous diffusion formulations.

The results⁵ of the first in a series of diffusion experiments to test the use of wind statistics as diffusion parameters at the National Reactor Testing Station are shown in Figure 3, which illustrates the method of determining the point of maximum concentration from the standard deviation of the vertical wind. Figure 4 shows a comparison between predicted and measured concentration values, using wind statistics as diffusion parameters. The new and attractive aspect of this technique is that it uses physical measurements of the wind, and one needs draw no vague inferences as to the appropriate values of indirect parameters in order to apply these data.

A bi-directional wind vane can be used to obtain the required wind statistics. This particular

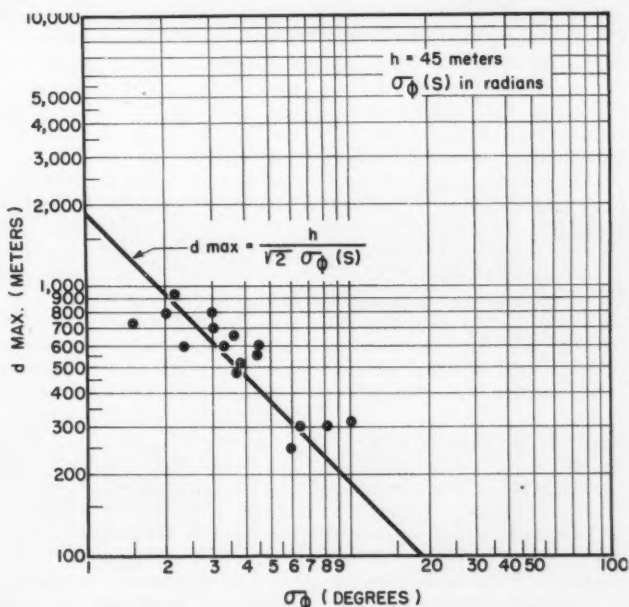


FIGURE 3. Point of maximum concentration vs. vertical wind.

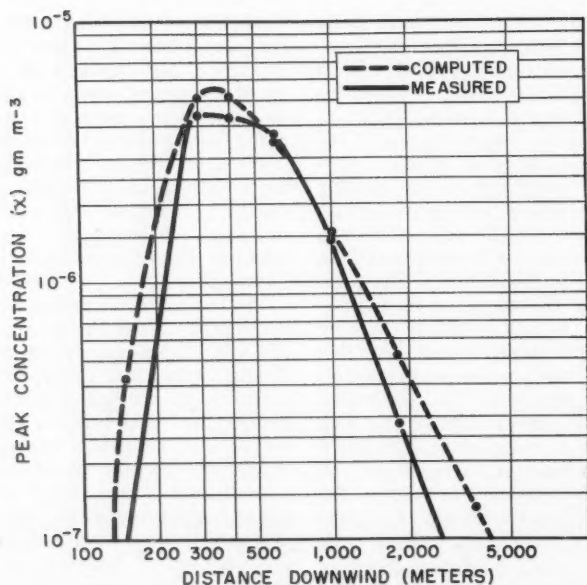


FIGURE 4. Prediction of concentration values using wind statistics.

type of instrument measures both horizontal and vertical winds, and thus provides the basic input data for calculation of the spread of material in both the horizontal and vertical directions. To assist in the handling of the data from such an instrument, a "black box" of electronic circuitry which automatically provides the required statistics (such as the variance or the standard deviation of wind fluctuations) has been constructed in England and other versions are being tested in this country. In addition, it is a simple matter to prepare a computer program for the handling of vast amounts of such data. We have found that the handling of 4800 observations of wind data for more than 100 different statistical calculations occupies about one minute of IBM-7090 time. A field installation of wind instrumentation, including a bivane, is currently being used at the National Reactor Testing Station in conjunction with their diffusion tests.

In order to arrive at the point where these wind data could be used in the manner shown it was necessary to perform tracer experiments in the field. Such experiments are usually time consuming and costly, requiring as they do, many sampling points, an appropriate tracer material, and most importantly, a quantitative analysis of the tracer. Recent developmental work by the Weather Bureau staff at the National Reactor

Testing Station,⁶ building on a suggestion by Robinson,⁷ has produced a successful field technique using uranine dye which, when atomized from a water solution, becomes a very small particulate in the micron range that can be collected readily with standard high volume samplers. What makes this technique so useful is that quantitative measurements of the tracer are accomplished by relatively simple techniques involving only the washing of the filters and measuring the fluorescence of the resulting solution with a sensitive fluorometer. The technique is quite rapid; two men can completely process about 100 samples in an 8-hour day, and sensitivity of the technique is from 10^{-9} to 10^{-10} grams per cubic meter.

While such a sophisticated tracer technique is necessary to test meteorological diffusion theories and which may be useful where there is a significant airborne waste problem, generally it is neither possible nor desirable to establish such an elaborate program. The use of visible tracers in determining atmospheric transport has been common for many years, and such work has the advantage that one does not have to guess where the cloud of airborne material is moving. Recently Gifford⁸ has formulated a mathematical basis for quantitative determination of diffusion parameters based only on observations of the geometry of smoke plumes. Culkowski⁹ has de-

veloped photographic techniques for recording smoke plumes which permit an operational application of this technique. Figures 5, 6, and 7 illustrate what can be done with time exposure photography to determine dispersion over different time intervals. Figure 5 shows the smoke from a standard Chemical Corps smoke pot. The photograph was taken at an exposure time of

1/100 second. Figure 6 shows smoke from the same source; the exposure time was ten seconds, and here we see how the violent gyrations of the plume are beginning to average out. Figure 7 shows the same plume, but the exposure time is eight minutes. Now we see that the visible portion of the plume begins to resemble the classic elliptical shape. Comparisons of the diffusion

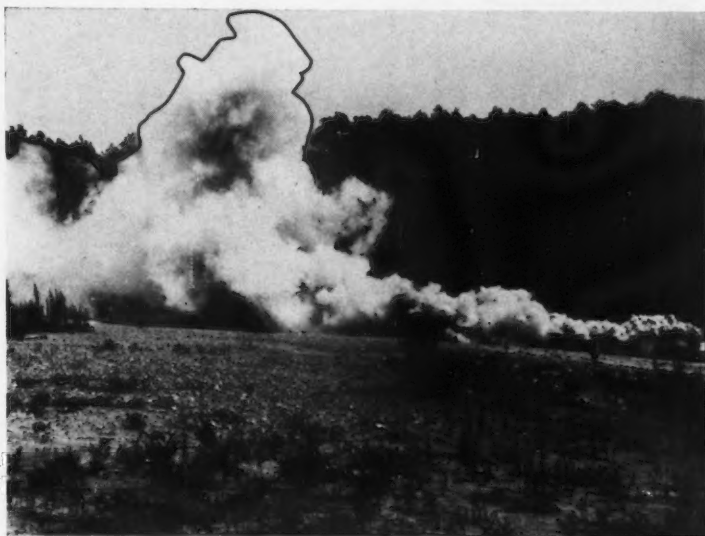


FIGURE 5. Smoke tracer experiment—exposure time 1/100 sec.



FIGURE 6. Smoke tracer experiment—exposure time 10 sec.

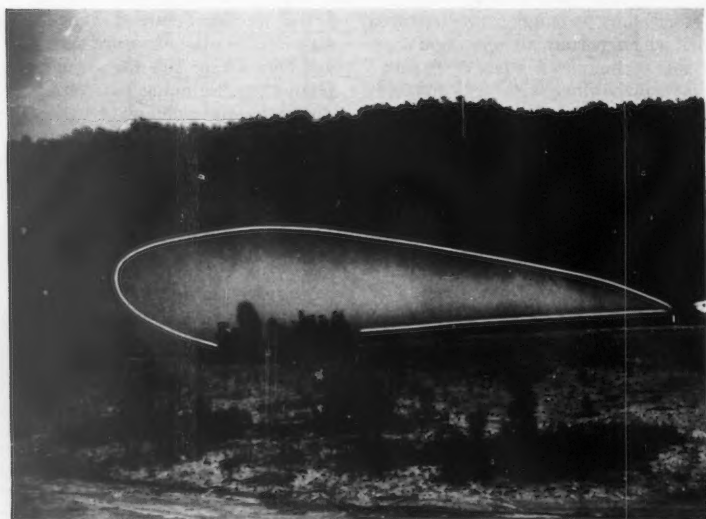


FIGURE 7. Smoke tracer experiment—exposure time 8 min.

coefficients determined from smoke releases accompanying quantitative tracer tests showed an almost one-to-one comparison. Using pictures such as these and the mathematical techniques of Gifford, it is possible to determine in a short time quantitative values of diffusion parameters for individual sites. Obviously this can be done for different times of the day, for different wind directions, and for different seasons of the year.

It appears that it should be possible to utilize this technique at night by illuminating the plume. It has proved possible to do such photography on clear moonlit nights through the use of the proper film.

Once one has related wind statistics to quantitative diffusion parameters determined from smoke or other tracers and has continuing records of the wind, it is possible to quantitatively

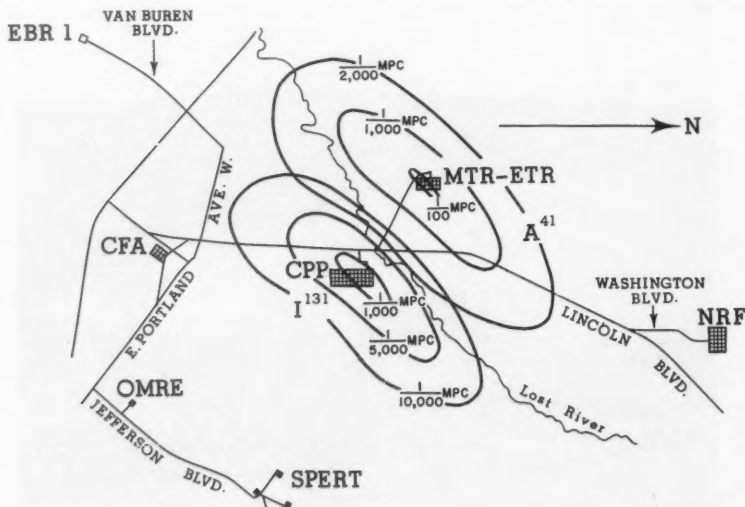


FIGURE 8. Average annual concentration isopleths.

compute the spatial distribution of concentration over various periods of time. A numerical technique for doing this has been developed at Oak Ridge⁹ and an example of this technique applied at the National Reactor Testing Station¹⁰ is shown in Figure 8, where isopleths for two different sources have been computed over

a one year period. When more than two or three such sources are involved, the computation becomes extremely laborious. In connection with research in urban air pollution a mathematical and computer model has been developed¹¹ which will handle more than 200 separate source locations and calculate their cumulative effect at

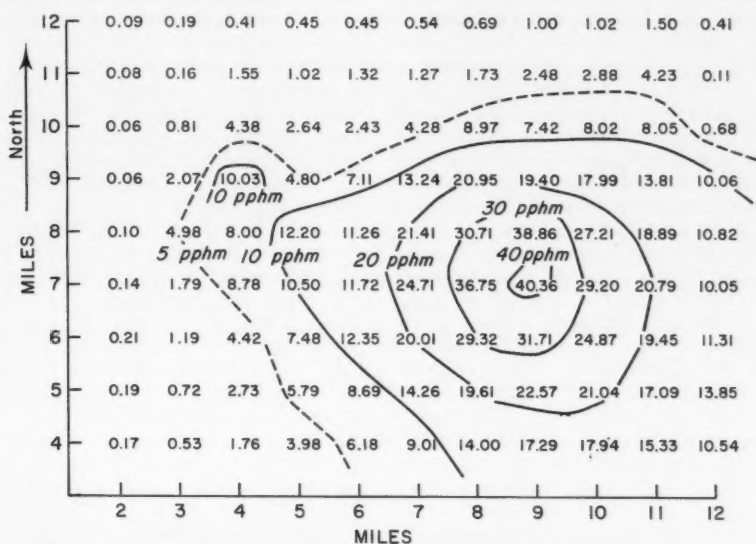


FIGURE 9. Machine computations of SO_2 concentrations from many sources.

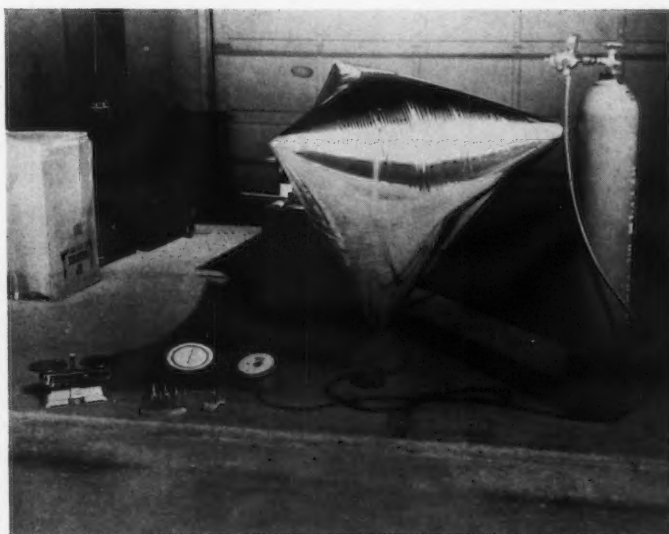


FIGURE 10. Constant volume balloon (tetroon).

more than 100 points. Figure 9 is an illustration of the machine output of this program. The horizontal and vertical scales are in miles and the isopleths are concentration of sulfur dioxide in parts per hundred million. This represents cumulative air concentrations from many sources at these numerous points. The isopleths of air concentration on this particular illustration were prepared by hand, but it is possible to

escape this labor since the machine can be programmed to draw the lines. Relatively short period concentrations can be computed, say over two hours, for each of these locations, and then the values summed to get weekly, monthly, or annual concentrations or doses. The volume of such computations is obviously enormous. If one has 200 sources, required concentrations at 100 points every two hours, and wanted to obtain

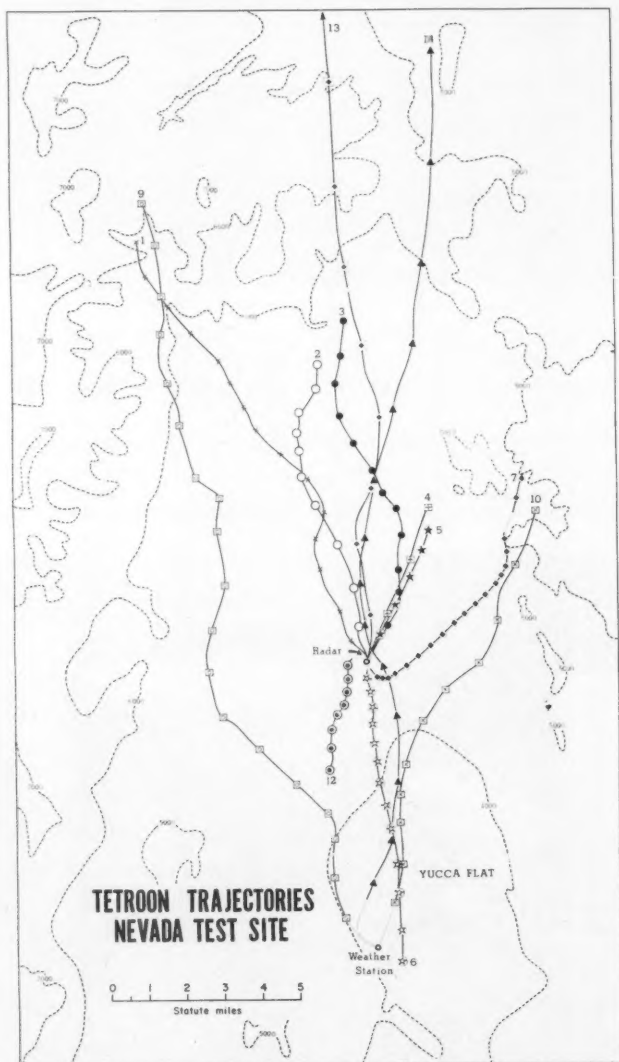


FIGURE 11. Tetroon trajectories, Nevada Test Site.

a monthly summation, then it would require 7,200,000 separate diffusion calculations. If one is very fast and can do one such computation every second, and do it 24 hours a day, it would still take 88 days to perform the work by hand (or every month you would fall two months behind). Instead the computations were made on an IBM-7090. A complete set of calculations for a day can be completed in about two minutes, so that a month's work can be done in an hour.

For many problems, however, these general computations are insufficient and one wishes to know precisely the direction in which any airborne effluent has travelled. For such problems, and particularly over distances between 10 and 100 miles, our experiments have proved the feasibility of tracking small constant volume balloons with radar.^{12, 13} The balloon that has been used is shown in Figure 10. These "tet-roons" (the shape is approximately that of a tetrahedron) are constructed of 2 mil plastic and have the ability to contain helium at appreciable superpressures with a minimum of stretching and practically zero leakage. Examples of trajectories that can be obtained are shown in Figure 11. These data were obtained at the Nevada Test Site of the Atomic Energy Commission at Las Vegas, Nevada. The observations were taken in the Yucca Flat area. Some

of the balloons were released from near the radar in the center of the figure; others, and this is a more interesting technique, were released some eight miles upwind of the radar. This permits using the same radar to obtain trajectories which originate from many points, for example, at more than one reactor. Numerous other experiments have been made, along the Atlantic Coast at Cape Hatteras and at the Wallops Station of NASA for example, but these particular trajectories (Figure 11) were chosen for illustration since the terrain over which the balloons flew at Las Vegas is extremely irregular. There were doubts as to whether or not it would be possible to use the radar tracking in mountainous terrain, but trajectories were obtained which were almost invariably more than 10 miles in length, and some of the flights could be tracked for as much as 40 miles. Figure 12 shows a height profile of three of the tetroon flights, and illustrates the dramatic difference between the vertical air motions from day to night. During the day vertical currents extended up and down through several thousand feet, as shown by flights 9 and 14. In contrast flight 6, at night, showed only the slightest of vertical motion. Thus, in addition to trajectories we are able to obtain excellent turbulence statistics at relatively great distances.

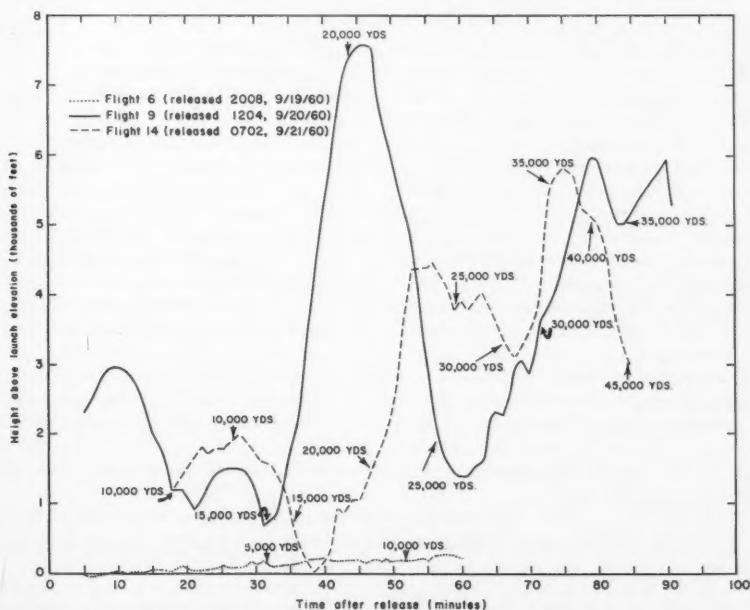


FIGURE 12. Vertical oscillations of tetroons at Las Vegas, Nevada.

Tests have also been made with these balloons off the southern California coast. These are particularly interesting experiments since no other means exist for obtaining air trajectories over water. One tetron was tracked for about eleven hours.

The tools and techniques here described are not the ultimate solution to the management of airborne wastes. Most importantly, there needs to be frequent contact between the staffs concerned with health and operating responsibilities at nuclear sites and the meteorologists. Such interchange will insure that a meteorological program can be tailored to fit the requirements of a particular facility. More importantly, new waste management problems will be identified and anticipated, and the meteorological research required to provide working answers to these problems can be arranged.

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TOXICOLOGY OF AIR POLLUTION

A LARGE-SCALE ANIMAL STUDY designed to determine the possible effects of air pollution on human health will be undertaken at the Wayne State University College of Medicine in Detroit. Under a \$190,000 Public Health Service contract, researchers at Wayne State will study more than 4,000 experimental animals — rats, guinea pigs, rabbits, and mice — with particular emphasis on pulmonary function, length of life, blood studies, and pulmonary pathology. The animals will be divided into three groups, the first of which will breathe exhaust-contaminated air pulled in directly from a heavily traveled street through a large duct especially constructed for the study. The second group will be exposed to the normal urban air present in the laboratory. The third group will be housed in a room supplied with specially cleansed air, from which almost all impurities will have been removed. Many of the animals will be kept under study during their entire lifetimes; others will be sacrificed to permit studies of changes in tissues of the lungs and other vital organs.

Among the common air pollutants which will be measured continuously, using automatic electronic instruments, are carbon monoxide, total hydrocarbons, oxides of nitrogen, total oxidants, sulfur dioxide, carbon dioxide, and inorganic particulates.

Additional studies will involve human subjects exposed to conditions comparable with those of the animals.

In the study Dr. Arthur J. Vorwald, Professor and Chairman of the Department of Industrial Medicine and Hygiene at Wayne State, will employ for the first time a technique which will permit measurement of the cardio-pulmonary function of small laboratory animals. It is believed that these measurements will add important data to the study results. The animals in the study will be subjected to a greater variety of physiological, biochemical, and histological observations than have been used in most tests of this kind.

Methods for Estimating Oxides of Nitrogen Emissions from Combustion Processes

PAUL F. WOOLRICH*

*Robert A. Taft Sanitary Engineering Center, U.S. Public Health Service,
Cincinnati 26, Ohio*

Methods for estimating emissions of oxides of nitrogen from combustion processes have been developed. Nitrogen oxide formation is related to temperature or heat supplied in the combustion process; and temperature or heat supplied is related to exhaust gas volume as a function of per cent CO_2 in the exhaust gas. The formation of nitrogen oxides is related to fuel consumption by a power function which is linear on a log-log plot. Equations and nomographs have been derived by means of which the pounds of nitrogen oxides per hour emitted from a combustion process may be estimated.

NITROGEN oxides are always present in the exhaust products discharged from combustion processes. Nitric oxide is the primary nitrogen oxide formed during combustion of organic fuel with air, although other nitrogen oxides may also be present. To determine the total amount of nitrogen oxides formed during combustion, an analytical method that permits measurement of nitric oxide as well as the small amounts of other nitrogen oxides present in the exhaust gases must be used. The phenoldisulfonic acid method¹ which was used in this work meets this requirement but does not distinguish between the individual nitrogen oxides. The data are, therefore, reported as total oxides of nitrogen expressed as nitrogen dioxide and generally termed " NO_x ".

The formation of nitrogen oxides during combustion has in general been found to be related to process exhaust volume and heat input by an exponential function. Fuel consumption can rarely be determined directly as it is usually not metered to the combustion equipment. However, if the volume of exhaust products and the carbon dioxide concentration have been determined, the consumption of a known fuel can be calculated. Carbon dioxide concentration is inversely related to the volume of exhaust product at a given fuel consumption. Further, the stoichiometric exhaust product volume from the combustion of all fuels, in sufficient quantity to

provide the same heat release, is approximately the same. Therefore, the hypothesis was established that the emission of oxides of nitrogen from a given heat input of fuels will be approximately the same when the exhaust product volume is adjusted to 12% carbon dioxide.

Utilizing data collected from a joint Los Angeles County Air Pollution Control District, California Department of Public Health, U. S. Public Health Service Study of Emissions of Oxides of Nitrogen from Stationary Sources in Los Angeles County,² a method for generalized estimation of oxides of nitrogen emission from gas and oil combustion processes has been developed empirically. The sources include oil and gas-fired household appliances, heaters of all types, ovens, kilns, metallurgical furnaces, small to medium-sized boilers and steam-generating power station boilers. The data from these approximately 1000 samples of combustion equipment effluent were evaluated to determine the relationships among heat input, volume of exhaust products, carbon dioxide concentration (wet conditions) and the pounds of nitrogen oxides emitted per hour at the time of sampling.

The empirical plot of pounds of oxides of nitrogen emitted per hour versus the volume of exhaust products in SCFM adjusted to 12% carbon dioxide was found to be linear. Figure 1 illustrates the spread of test data utilized to determine the experimental slope and intercept, as determined by method of least squares to provide the geometric mean. Statistically, the 95% confidence interval of the logarithm of the slope and the intercepts are 3.00 ± 0.13 and 0.84 ± 0.35 , respectively. More carbon dioxide is formed

Presented at the Twenty-second Annual Meeting of the American Industrial Hygiene Association, Detroit, Michigan, April, 1961.

* Present address: Occupational Health Research and Training Facility, Division of Occupational Health, U.S. Public Health Service, Cincinnati 2, Ohio.

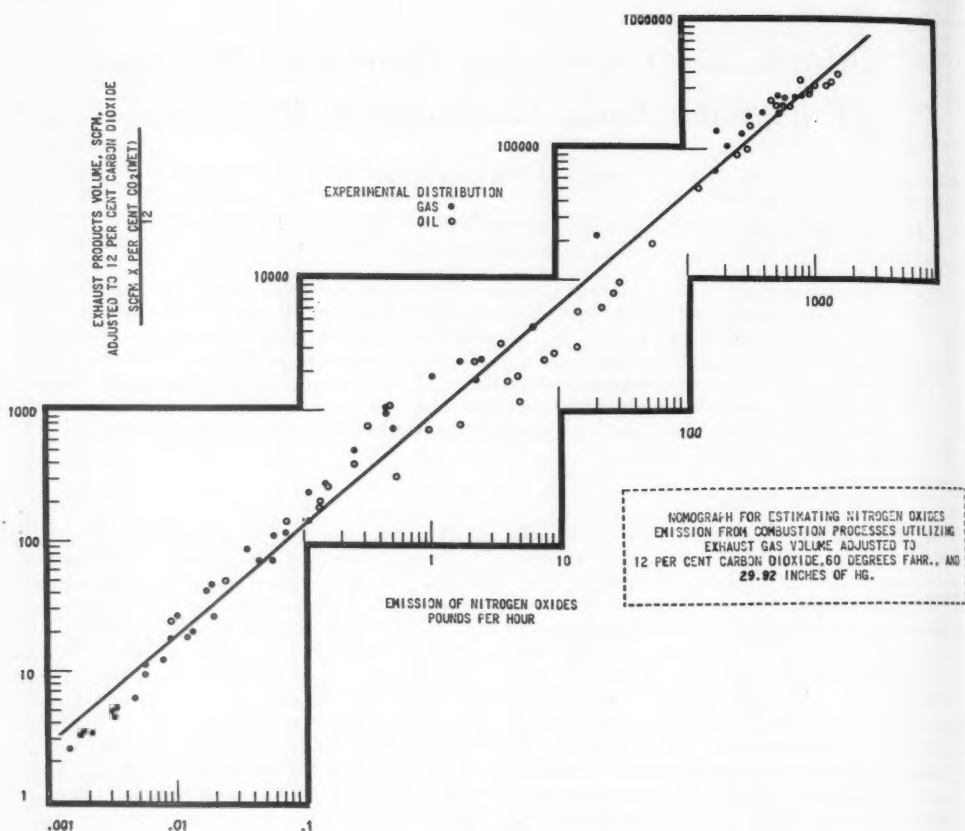


FIGURE 1.

in combustion of oil than in combustion of gas; therefore, the adjusted SCFM will be higher for oil. Thus, this data show that a given heat value of oil will produce more oxides of nitrogen than the same heat value of gas. The theoretical flame temperature for oil is about 200°F higher than that for natural gas, which would indicate a greater production of NO_x by oil. Expressed mathematically, the emission of nitrogen oxides in pounds per hour from combustion processes emitting variable quantities of exhaust products adjusted to 12% carbon dioxide is:

$$\text{pounds NO}_x = \left[\frac{\text{SCFM} \times \% \text{CO}_2}{938 \times 12} \right]^{1.18}$$

Where SCFM = measured or computed exhaust volume, adjusted to 60°F and 29.9" Hg.

% CO_2 = per cent by volume of CO_2 (wet conditions) in exhaust products.

A second empirical relationship relating nitrogen oxide emissions to heat input of the fuel can also be developed using the information previously obtained. From Figure 1 it is shown empirically that 938 SCFM of flue gas corrected to 12% carbon dioxide produces one pound of oxides of nitrogen per hour. Based on the volume of carbon dioxide formed by combustion of the fuel, the heat input for the production of one pound of nitrogen oxides may be calculated as follows:

For gaseous fuels:

heat input for 1 lb NO_x /hr

$$= \frac{(938 \times 60 \times 0.12)}{\Sigma n C_n H_m} H_v$$

Where $(938 \times 60 \times 0.12)$ = volume of CO_2 formed per pound of NO_x produced.

ΣnC_nH_m = volume of CO_2 produced by the combustion of the various hydrocarbons in one cubic foot of fuel gas.

H_v = heating value of the fuel in Btu/cf.

For liquid and solid fuels:

heat input for 1 lb NO_x /hr

$$= \frac{(938 \times 60 \times 0.12)}{31.6 \times C} H_v$$

Where C = % by weight of carbon in the fuel.

31.6 = number of SCF of CO_2 produced by the combustion of one lb of carbon.

H_v = heating value of the fuel in Btu/lb.

For the analyses indicated in the following table, the intercepts at one lb of NO_x per hour equal, for natural gas 6.60×10^6 and for fuel oil 4.44×10^6 .

Gas		Oil	
CH_4	88.12%	C	87.15%
C_2H_6	7.48	H_2	9.42
C_3H_8	2.22	S	1.46
C_4H_{10}	0.27	O_2	1.01
N_2	1.48	N_2	0.90
CO_2	0.43	H_2O	0.02
H_v	1091 Btu/cf	Ash	0.04
		H_v	18135 Btu/lb.

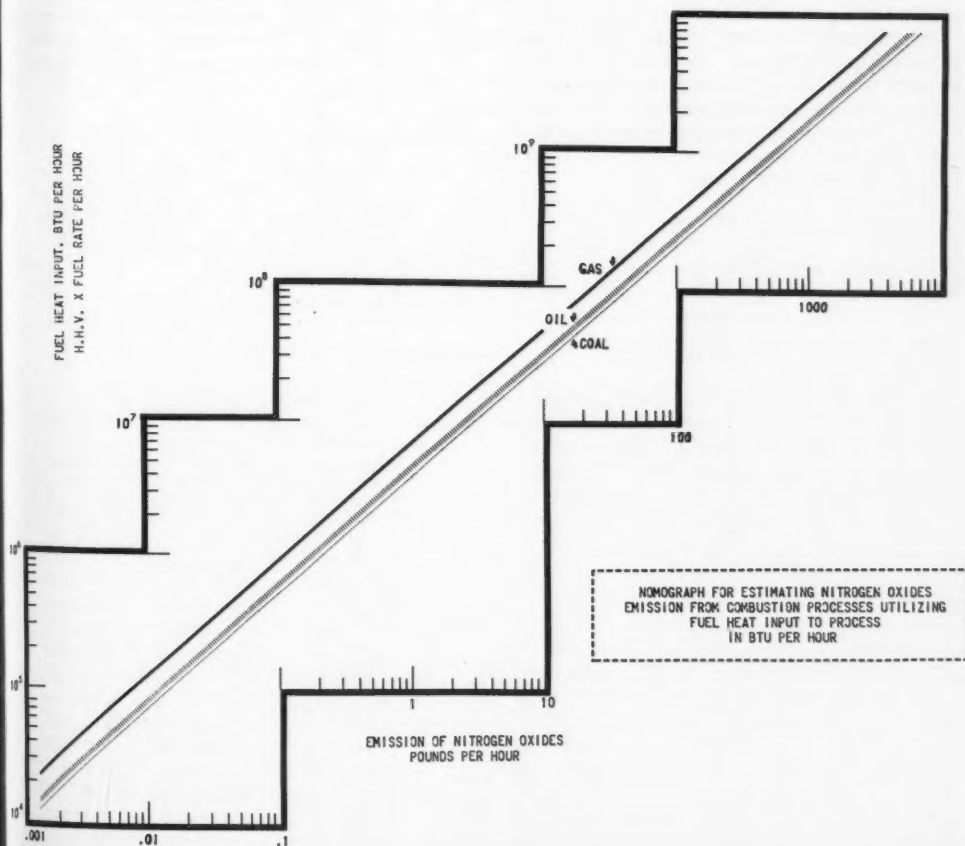


FIGURE 2.

Using these values as intercepts on the logarithmic plot of emission of oxides of nitrogen in pounds per hour versus fuel heat input in Btu/hr and the reciprocal slope of 1.18 previously developed, the mathematical relationship between heat input and NO_x emission is:

$$\text{pounds NO}_x/\text{hr} = \left[\frac{\text{Btu/hr}}{6.60 \times 10^6} \right]^{1.18}$$

$$\text{pounds NO}_x/\text{hr} = \left[\frac{\text{Btu/hr}}{4.44 \times 10^6} \right]^{1.18}$$

Where Btu/hr = total heat input from fuel per hour.

The ultimate analysis of the fuel oil and natural gas burned during collection of this data is known. By including the ultimate analysis of a typical Pocahontas coal, equations relating emission of nitrogen oxides from various heat input of this fuel is attainable. Equations for gas and oil have as their basis of derivation extensive experimental test data of equipment utilizing natural gas or fuel oil. The equation for coal is based on the stoichiometric relationship of exhaust volume and ultimate analysis of coal in lieu of experimental data obtained specifically for this fuel. The ultimate analysis of this fuel is (% by weight):

	Coal
C	83.39%
H ₂	4.56
O ₂	5.05
N ₂	1.03
S	0.64
Ash	5.33
H.V. 14635 Btu/lb.	

The slope and intercept of the plot representing heat input versus NO_x emissions for the above typical coal are 0.846 and 3.8×10^6 . In this way, the curve for any fuel for which the ultimate analysis is known can be theoretically calculated.

The log plot of these relationships is shown

in Figure 2. It will be noted that the emission rate per million Btu is greater as heat input increases. In conducting an emission inventory, therefore, it is important to know the size of combustion equipment being considered. For example, if all other factors are equal, a million KW/hour boiler will emit more NO_x than five 200,000-KW/hour boilers.

A full discussion of the many variables affecting the production of oxides of nitrogen in combustion processes has been published in the Joint Report on the Study of Emissions of Oxides of Nitrogen from Stationary Sources in Los Angeles County.² However, when the techniques, as indicated in the reference, are used to establish the oxides of nitrogen emission from combustion sources, the only variable requiring compensation is that related to the increased heat input from preheated combustion fuel air mixtures such as are found in power station boilers. If the combustion air is preheated, the emissions as determined by the above relationships should be multiplied by an average factor of 1.14 for temperature ranges of 420°F. to 650°F., and excess air ranges of 12% to 65%.

Acknowledgments

The cooperation of personnel of the Los Angeles County Air Pollution Control District during preparation of this manuscript is gratefully acknowledged. The statistical evaluation of test data was augmented by the efforts of Mr. Merle E. Meyer, Assistant Sanitary Engineer, U. S. Public Health Service.

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An Incinerator for Wastes Containing Microcurie Amounts of Carbon-14

VICTOR E. LAVETTER

Bureau of Industrial Hygiene, Detroit Department of Health, Detroit, Michigan

A description is given of a small rotating drum incinerator designed for the quantitative ignition of a radionuclide whose combustion product is gaseous at normal temperature and pressure. Samples of the flue gases and mineral residues indicated that the system was safe for disposal of small animals containing microcurie amounts of radiocarbon. Flue gases from the special incinerator mix with those from a large municipal incinerator, and discharge through a chimney 150 feet high after the mixture passes through a wet fly ash collector.

Introduction

SEVERAL years ago a medical research group in Detroit changed over from the use of phosphorus-32 to carbon-14 labelled compounds for investigation into the etiology of cancer in rats and mice.

In experiments where four to five millicuries of phosphorus-32 were formerly injected per animal, 45 microcuries of carbon-14 were subsequently used. At one time inadequate allowance for radioactive decay of the phosphorus-32 resulted in an excessive concentration of radio-phosphorus in the incinerator ash. This was corrected by storage of the carcasses in a deep freeze unit until the radioactivity became negligible before incineration.

On changing over to the use of radiocarbon labelled compounds, it was decided that disposal in the institutional incinerator would be unsatisfactory for several reasons. First, the grate design was such as to permit unburned, or partly burned, material to reach the ash pit, so that some hazard would exist for personnel during transfer of the ash from the incinerator to receptacles, and subsequently to rubbish trucks. Second, isotopic dilution would be difficult to attain, at least in a sufficiently reliable manner to be safe, because of the smallness of the batches required by the size of the incinerator. Third, the chimney, although of adequate height for ordinary products of combustion, was considered too low for dependable diffusion of flue gases containing unknown concentrations of radioactive material either as gases, or as soot. Fourth, the institution was located in an area of high population density, requiring the utmost caution lest undesirable meteorological conditions permit an excessive concentration of radio-

active material to enter nearby dwellings from the institutional incinerator chimney.

The problem of transferring radioactive wastes to a licensed disposal facility, as recommended, appeared to pose undue difficulty to the research group, and, after the existing deep freeze unit became filled to capacity, a second storage unit was acquired. When the second deep freeze unit became more than half-filled with radioactive small animal carcasses, with no solution to their ultimate disposal in sight, an appeal was made to the Detroit Bureau of Industrial Hygiene for permission to incinerate part of the accumulated material to relieve the congestion which could ultimately interfere with the progress of their research. In fact, the accumulated waste was found to exceed the quantity of radioactive carbon permitted under the Atomic Energy Commission license. Since the original premises for prohibiting such incineration were still valid, other solutions to the problem were considered.

The Bureau of Industrial Hygiene contacted the Municipal Engineer of Waste Disposal, explaining the need for an adjunct to a municipal incinerator which would quantitatively ignite all carbonaceous material, and subsequently dilute the products of combustion with those from an incinerator handling large quantities of stable carbon. After considerable experimentation, the Engineer of Waste Disposal developed the facility which is the subject of this presentation.

The City of Detroit operates several incinerator plants, and the Central plant was chosen to be used for this purpose because of its proximity to anticipated users. Another compelling reason for its selection was the fact that, as part of Detroit's long-range program of equipping munici-

pal incinerators with improved fly-ash collectors, the Central plant already had a wet fly-ash collector connected to one of its three units.

Design of Incinerator

Preliminary experiments with ordinary "wet" wastes indicated that such wastes tend to char, rather than ignite, in a stationary drum. This occurred even when temperatures used were high enough to cause the steel drum to deform. It was found necessary to construct a rotating drum of $\frac{1}{8}$ inch thick stainless steel, whose specifications would permit operating temperatures up to 2000°F.

The combustion chamber is 18 inches in diameter and 24 inches long. The end of the drum at which the loading and firing is done has a nine inch diameter opening, and the flue opening on the opposite end is seven inches in diameter. The nine inch disc cut out of the loading opening is used as a baffle just inside of the combustion chamber at the flue opening, to prevent direct blow through.

An outer drum of ordinary $\frac{1}{8}$ inch thick sheet steel, 26 inches in diameter and 32 inches long, is welded to the stainless steel combustion chamber by means of stainless steel sleeves at the

loading and flue openings. All welded construction is used. The 4-inch space between the combustion chamber and the outer drum is filled with castable low density refractory (Vermiculite in cement—40 pounds per cubic foot). The outer drum rests on rollers which cause the drum to rotate, with its axis sloping slightly downward one inch in 33 inches from the loading opening.

A gun type 100,000 BTU per hour gas burner is mounted on a sliding frame external to the 9-inch diameter loading-firing opening. When the gas burner is in firing position, a steel plate closes off the balance of the firing opening. In order to place material in the combustion chamber, it is necessary to shut off the gas burner and slide it back from the loading-firing opening, after which it is re-ignited and returned to the normal firing position.

A one-inch internal diameter stainless steel drain pipe is welded onto the lowest point of the combustion chamber to facilitate flushing of residues directly to the sewer after the unit has cooled, and without necessitating any handling of such residues. A gate type valve keeps the drain pipe closed during incineration. The rollers are driven by an electric motor, with interven-

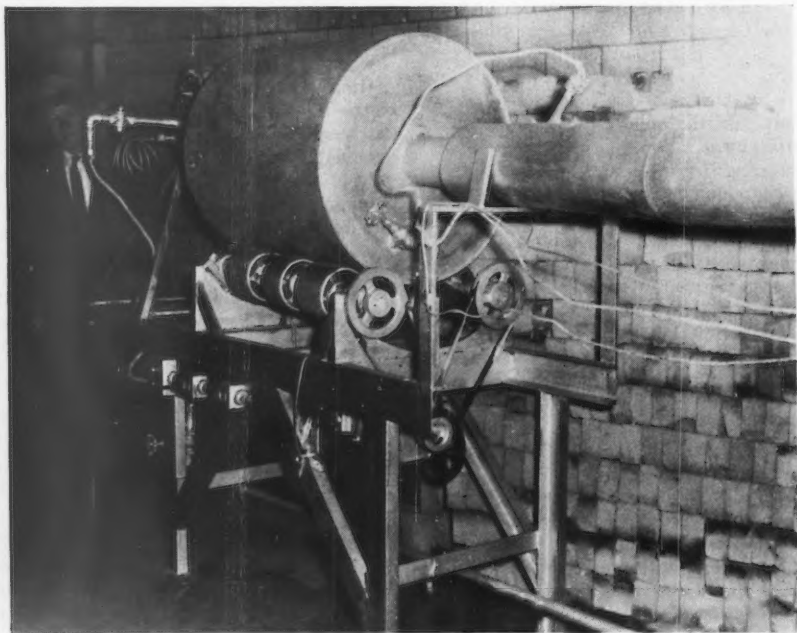


FIGURE 1. Back view of the special incinerator.

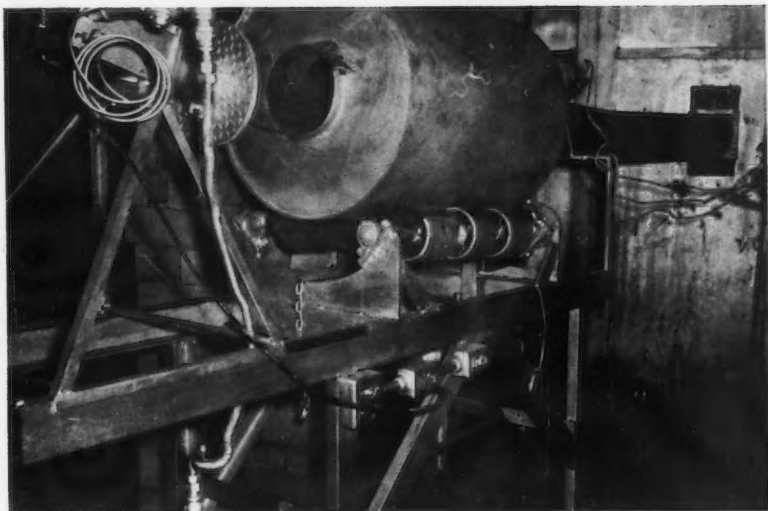


FIGURE 2. Front view of the special incinerator.

ing reduction gears and pulleys, so as to cause the drum to rotate at a rate of about two revolutions per minute.

A thermocouple is located just inside of the 7-inch diameter stainless steel rotating flue, at the point where it discharges into the stationary 9-inch diameter galvanized iron flue. The one-inch annular space between the rotating and stationary flues admits room air into the flue, causing dilution and cooling of the flue gases prior to discharge into the wet fly-ash collector.

The precipitation chamber of the wet fly-ash collector is 16½ feet wide, 26 feet long and 27½ feet high, with the hot gases entering at the top of one corner from an incinerator handling 275 to 350 tons of combustible waste daily, and leaving at the bottom of the opposite corner after passing through a water curtain sprayed on the first of two sets of baffle plates. Eighty-four spray nozzles, contained in six headers, handle a total of 150 gallons of water per minute. A depth of eight inches of water is maintained in the bottom of the precipitation chamber, resulting in an average water retention time of about 14 minutes.

The breeching, 6 feet wide and 9 feet high, is at the base of the 150 foot high chimney, whose internal diameter is plumb at 8½ feet. A 6-inch firebrick target wall reduces the internal diameter of the chimney to seven feet two inches at the base, to a height of 45 feet. The negative pressure created by the chimney causes a linear inflow of air into all openings of the carbon-14

incineration system in excess of 300 feet per minute.

Operation of Incinerator

When small animal carcasses containing radio-carbon are to be incinerated, the rotating drum is preheated for 30 minutes to a flue temperature ranging between 1600 and 1800 degrees Fahrenheit. Addition of a 5-pound package of frozen small animals to the preheated combustion chamber causes the flue temperature to rise about 200 degrees Fahrenheit almost immediately, therefore the starting flue temperature is carefully controlled so as not to exceed 1800 degrees Fahrenheit. The time required to reduce five pounds of frozen small animal carcasses to a fine white ash was found to be about 25 minutes.

The residual ash in the combustion drum was found to contain less than 0.008% of the radioactivity contained in the animals at the time of incineration. Rinsing of the drum has been found to be unnecessary because of the small amount of residue from each incineration. The ash appears to be sufficiently light as to be ultimately swept into the flue gases, and subsequently washed to the sewer with the liquid effluent from the precipitation chamber.

The permissible rate of incineration was calculated on the basis of 0.1 micro-microcurie maximum permissible concentration per cubic centimeter of air; air density of 0.075 pounds per cubic foot; 3.5 pounds of air used, minimum,

per pound of total waste; and a minimum of 275 tons of waste handled per day; and this turned out to be 50 microcuries per minute for this system. The calculated permissible rate of incineration for isotopic dilution, based on 0.1 microcuries maximum permissible concentration of radiocarbon per gram of stable carbon, and 0.3 pounds of carbon per pound of waste, turns out to be 5200 microcuries per minute. By using the figure for air dilution alone the safety factor becomes two orders of magnitude when compared with the permissible rate for isotopic dilution.

Gas samples collected in the nine-inch flue indicated a concentration of 0.084 micro-microcuries per cubic centimeter at an incineration rate of 1.35 microcuries per minute. This was well within the permissible limit for direct discharge to the atmosphere, without further dilution. At higher rates of incineration, samples should be collected either in the chimney, or in the breeching, to verify the predicted concentration.

This facility is not licensed as a disposal agency for radioactive wastes, but it is made available for the use of Detroit licensees who obtain Atomic Energy Commission approval to dispose of microcurie amounts of radiocarbon in this facility. Disposal of radionuclides other than carbon-14 in this facility is not currently being permitted. Should the need arise, it is conceivable that other radionuclides whose products of combustion are gaseous at normal tem-

peratures and pressures, might be disposed of in this facility. It is probable that tritium can be safely incinerated in this facility.

Summary

In summary, a facility has been described for the incineration of wastes containing microcurie amounts of a long lived radioactive isotope (carbon-14) whose combustion product is gaseous at normal temperatures and pressures. Assay of the residual ash and gas samples collected in an intermediate flue indicate that the procedure is safe at the rate of incineration used. Calculations indicate that the method is probably safe for substantially higher rates of incineration.

Acknowledgments

Acknowledgment is gratefully given to Mr. Theodore Winkler, Engineer of Waste Disposal, for designing, experimenting with, and building the facility described; to Doctor William G. Fredrick, Director of Industrial Hygiene, for initiating the project, and for his valuable guidance; to Mr. Alphonse J. Kaimala, Associate Industrial Hygienist, for material assistance in arranging for and sampling of the flue gases; to Doctor Richard F. Skonieczny for preparation of sampling reagents and recovering the carbonaceous precipitates; and to Doctor Donald Willard of the Occupational Health Staff at Wayne State University's Medical School, who permitted the use of his Department's liquid scintillation counter for assay of samples.

RADIOACTIVITY STANDARD MATERIALS

THE NATIONAL BUREAU OF STANDARDS has announced that two additional standard samples of radionuclides — iron-55 and promethium-147 — are being issued. These two standards now available, result from the Bureau's continual program to establish new and improved radioactivity standards. Both of the standards have wide applications for medical and industrial laboratory purposes. Iron-55, an electron-capturing nuclide with a half-life of 2.7 years, decays by x-ray and Auger-electron emission directly to the ground state. It is used extensively as a tracer both in biological and industrial research. Promethium-147 is a pure beta emitter utilized for generation of low-energy x-rays and energy calibration standards for x-ray and gamma-ray spectrometers. It has a half-life of 2.5 years. In addition to other research applications, this radionuclide is incorporated in luminescent markers and is being considered for use in nuclear batteries.

Both of the standard samples may be ordered under the general licensing provisions of the Atomic Energy Act of 1954. The iron-55 standard sample No. 4929 and the promethium-147 standard sample No. 4940 may be purchased for \$36.00 and \$40.00 respectively. Both samples are in solution form (approximately 3 ml), and have nominal activities of 5×10^4 dps/g. The iron-55 solution contains 1 $\mu\text{g/ml}$ FeCl_3 as carrier, while the promethium-147 solution is carrier free. All orders for the radioactivity standards should be addressed to Miss Elizabeth M. Zandonini, Radioactivity Standard Samples, Radioactivity Section, National Bureau of Standards, Washington 25, D. C.

Health Aspects of the Commercial Melting of Radium Contaminated Ferrous Metal Scrap

R. H. STARKEY, S.M., J. A. QUIGLEY, M.D.,
and J. W. McKELVEY

*Feed Materials Production Center, National Lead Company of Ohio,
Cincinnati 39, Ohio*

Procedures and tests are described whereby it is shown that radium in ferrous metal scrap is virtually all removed in the slag on remelting. The smelting of radium-contaminated ferrous scrap can be performed with safety for the workers by use of proper ventilation and other simple precautions. The purified ferrous metal will not constitute a radiation problem to health from subsequent uses of the metal.

Introduction

THE Feed Materials Production Center (FMPC) is one of the two U. S. Atomic Energy Commission (AEC) owned plants operated primarily for the purpose of supplying uranium metal fuel elements for the plutonium production reactors. At the FMPC, located approximately 20 miles northwest of Cincinnati, Ohio, and operated by the National Lead Company of Ohio (NLO), uranium ores and concentrates are received and processed into metal fuel elements. For the first seven years of operation, from 1951 through 1958, a portion of the raw materials was received as pitchblende ores which contained up to 600 mg of radium per ton. These ores were received in 30 and 55 gallon steel drums. Thus, over the years many empty radium contaminated drums were accumulated. In addition to the ore drums, approximately 15,000 drums of radium bearing residues from radium extraction processes were shipped from the Lake Ontario Ordinance Works, Niagara Falls, New York and the Mallinckrodt Chemical Works, St. Louis, Missouri for storage at the FMPC. At the completion of the processing of these radium bearing ores, much of the process equipment was removed and scrapped. This bulk scrap added to the ever growing pile of radium contaminated scrap iron. A total of approximately 350 tons of radium contaminated scrap was accumulated in all. The drums and bulk scrap were cleaned of all loose contamination and the drums baled to reduce their bulk and the area needed for storage.

The AEC Manual for prime contractors (Chapter 5182) covers the disposal of uranium

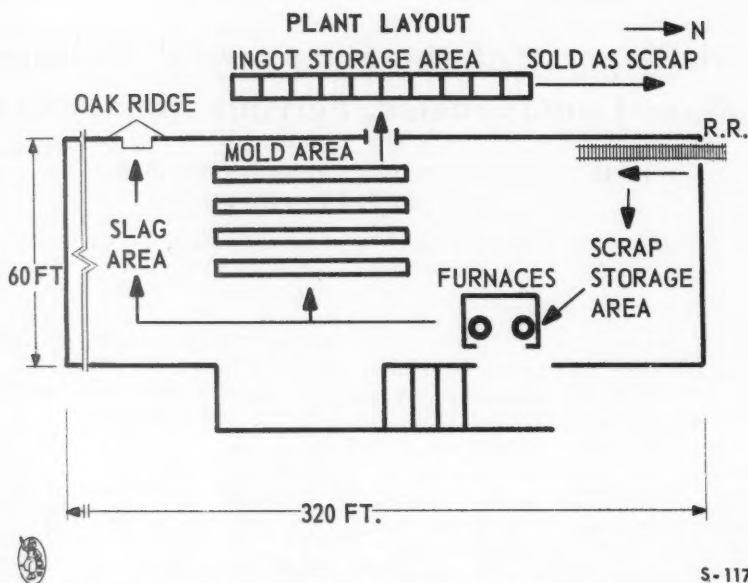
contaminated ferrous scrap, but does not, in any way, include the disposal of any radium contaminated scrap metals. Thus, this scrap was stored at the FMPC awaiting some approved method of disposal. As stated earlier, the AEC Manual permits the open sale of ingots obtained from the smelting of uranium contaminated scrap, 6000 tons of which were being disposed of by the FMPC at the time the radium contaminated drums were being baled.

It was thought that the radium would react to smelting in much the same manner as uranium, that is, it would virtually all be removed in slag. Previous papers presented by representatives from Health and Safety Laboratory (HASL), Union Carbide and our own group cover the health hazards of melting uranium contaminated scrap and each describes the path taken by the uranium in various test melts.^{1, 2, 3} Therefore permission was requested of the AEC to conduct a test melt with 40 tons of the baled drums for the purpose of determining whether or not the radium would react as we thought and be removed in the slag to a sufficient degree to permit open sale of the resultant steel ingots. AEC approval was granted and the test was conducted on October 7-8, 1958, in the same mill in which the uranium contaminated scrap was being processed. The 40 tons of radium contaminated scrap were blended with 20 tons of uranium contaminated scrap for this test melt. This paper describes only this test, and the results obtained therein, inasmuch as the uranium melt was covered in a paper presented at this conference in 1959.³

Methods

Figure 1 shows the approximate floor plan and size of the area involved in the melting of

Presented at the Twenty-second Annual Meeting of the American Industrial Hygiene Association, Detroit, Michigan, April 12, 1961.



S-1173

FIGURE 1. Layout of plant area involved in the melting of contaminated scrap.

the uranium and radium contaminated scrap. The arrows indicate the material flow through the mill. The scrap entered the mill at the scrap storage area and was transported by crane into the furnace. Following the melting operation, the ingots were poured in the mold area and then stored outside on the west side of the mill. The resultant slag was stored in the slag storage area until it was shipped to Oak Ridge for disposal.

The arc furnace used had a removable top section containing carbon electrodes. The bottom section was charged with approximately two ton charges of scrap bales and each heat consisted of three or more charges. Molten metal from each heat was poured into a ladle by removing the top of the furnace and tilting the bottom section. The molten metal was transported in the ladle by an overhead crane, to the ingot molds. The metal was poured into molds through a hole in the bottom of the ladle with the flow being controlled by a stopper rod. A metal test spoon was used for taking three metal samples from each melt, at approximately the beginning, middle, and end of each pour. The slag which was retained in the ladle was then dumped into a slag thimble where it was permitted to solidify. Three samples also were taken from each slag pour.

Air dust samples were taken at various locations by drawing air at 20 lpm (liters per min-

ute) through a Whatman #41 filter paper. In this case all air samples taken were of a "general air" type, although they are considered to be representative of individual exposures. Collection of samples from the breathing zone was not practical because of the wide area of movement by the operators and the safety hazard involved with sampling in the immediate area of the furnace during the charging and pouring operations.

The alpha activity in the air dust samples was determined by standard counting methods using scintillation counters. For evaluation purposes all alpha radioactivity was assumed to be coming from radium inasmuch as it has an appreciably lower Maximum Allowable Concentration (MAC) than does uranium, the other major alpha emitting contaminant present.

Determination of Radium by the Radon Method

Radium-226 decays by alpha emission to radon-222 which itself decays by alpha emission and has two very short half-life alpha emitting daughters. Since radon-222 is a gas, it can be separated from the sample and collected. The alpha activity of the radon-222 and its daughters is determined and related to the original radium-226 content of the sample.

The sample is put into solution, diluted to 150 ml and transferred to a "radon growth flask." All radon is then flushed out by lightly

boiling the solution and flushing with nitrogen gas. The flask is sealed and the radon is allowed to "grow in" (i.e. accumulate through radioactive decay) for a period of from 3½ to 24 hours, depending upon the expected approximate concentration of radium. The flask is then attached to a purification train which removes O₂, H₂O, and CO₂. The radon is allowed to flow with a carrier gas (nitrogen) through the evacuated train into an evacuated ionization chamber.

The alpha activity in the ionization chamber is then counted. From the alpha count, the weight of radium per gram of sample is calculated from the equation:

$$\frac{\text{gm Ra}}{\text{gm Sample}} = \frac{(\text{cps})}{2.22 \times 10^{12}(T)(S)(E)(3)}$$

where:

cps is the alpha count in counts per second,
 2.22×10^{12} is the radium decay factor in disintegrations per minute (dpm),

T is the growth time for radon in the sample (hours),

S is the weight of sample used (grams),

E is the efficiency of the detector expressed as apparent geometry, and

3 is a constant representing the contribution of radon daughters (two alpha-emitting daughters).

The apparent geometry of the ionization chamber is determined empirically by analyzing a sample with a known radium content.

Summary of Data

The average concentration of radium in the eight steel samples was 0.9×10^{-12} gram Ra per gram of steel. This is only about 0.06% of the average radium content of the slag samples which was 1470×10^{-12} gram Ra per gram of slag. The radium content of each of the eight steel samples is shown in Table I. Similar information is given for the slag samples in Table II. It should be remembered that each of the steel and slag samples is a composite of three individual grab samples.

Air dust samples were taken at five locations continuously throughout the entire test as shown in Figure 2. The average concentrations of these are shown in Table III and indicate an over-all average air dust level of 16×10^{-11} µc/cc. However the one location, E, although used for calculating the over-all average concentration, really misrepresents the over-all air contamination picture. The samples at this location were taken to attempt to determine the extent of the crane operator's exposure while loading the furnace. Since he moves back and forth the

TABLE I
Radium Content of Steel Ingots

Heat No.	ppm-U	gm Ra/gm steel
1	<1	$.20 \times 10^{-12}$
2	3	$<.01 \times 10^{-12}$
3	<1	$<.01 \times 10^{-12}$
4	<1	$.70 \times 10^{-12}$
5	1	1.60×10^{-12}
6	2	3.70×10^{-12}
7	<1	$.40 \times 10^{-12}$
8	2	$.60 \times 10^{-12}$
Average	<1.5	$<.90 \times 10^{-12}$

TABLE II
Radium Content of Slag

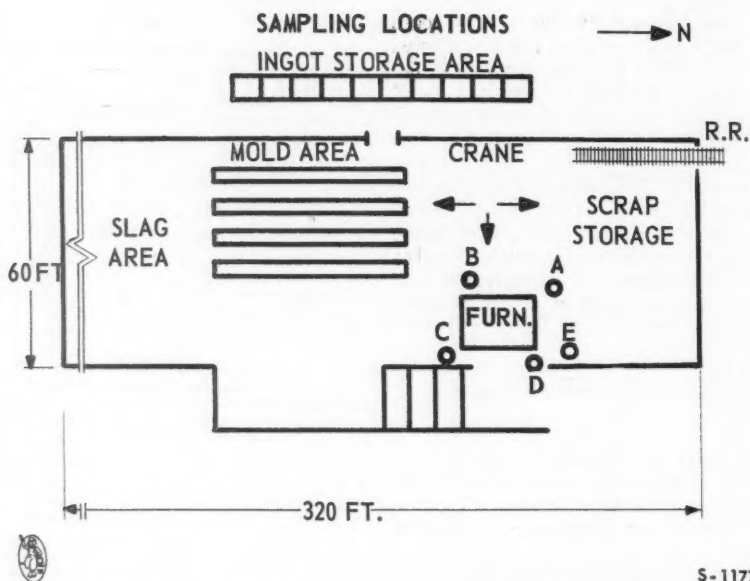
Heat No.	gm Ra/gm slag
1	1320×10^{-12}
2	920×10^{-12}
3	1440×10^{-12}
4	1870×10^{-12}
5	2700×10^{-12}
6	1470×10^{-12}
7	800×10^{-12}
8	1220×10^{-12}
Average	1470×10^{-12}

complete length of the mill in performing his work, he was exposed to this concentration for only a small portion of his working day. The remainder of his time was spent in other locations where the air dust concentrations should have been less than any shown in Table III. It was impossible to measure the air dust levels at these locations however, because of their inaccessibility. Disregarding this one location, the air dust levels averaged only 8×10^{-11} µc/cc or approximately 2.7 × MAC as listed in *NBS Handbook 69* for a forty-hour work week.

It is concluded that since respiratory protection was judiciously worn throughout this entire test, more than ample protection was afforded in reducing actual exposures to below the maximum allowable concentration as listed in *National Bureau of Standards Handbook 69*.

Discussion

This test melt with the radium contaminated scrap was conducted at the completion of a production melt of 6000 tons of uranium contaminated scrap. The operating procedures followed for this test melt were exactly the same as those used for the uranium contaminated steel melt. The test lasted for two days and was observed at all times by NLO Industrial Hygienists who also supervised the decontamina-



S-1177

FIGURE 2. During the processing of contaminated scrap air samples were collected at locations indicated by letters, A, B, C, D and E. Circles represent principal locations of workers.

TABLE III
Average Air Dust Levels

Location	$\alpha\text{-d/m}^3$	$\mu\text{c/cc}$
A	27	1.2×10^{-11}
B	37	1.6×10^{-11}
C	283	12.6×10^{-11}
D	381	17.0×10^{-11}
E*	1058	47.3×10^{-11}
Avg. with E	357	16.0×10^{-11}
Avg. excluding E	180	8.0×10^{-11}
MAC (NBS Handbook 69)	67	3.0×10^{-11}

* 35' above floor.

tion of all facilities following the test. All contaminated materials, furnace refractories, dirt, scrap, ladles and the slag from both the uranium and radium contaminated scrap melt were sent via government trucks to Oak Ridge for disposal.

Throughout the test melt all personnel working in the areas where scrap metal was being handled wore respiratory protection, whether or not they were working with contaminated metal. In addition, all personnel not directly involved in the test were sent home, in order that no undue exposures would result. This

was an added precautionary measure taken by the plant management. The respirators worn were of the Comfo-type equipped with ultra-filters.

In addition to respirators, protective clothing consisting of cotton duck coveralls, caps, and leather gloves was furnished to the mill employees for this entire test operation. The clothing was another added precautionary measure taken to minimize personnel exposures.

The usual locations of the men involved in this scrap melting operation are indicated on Figure 2 by black dots. Two 60-inch, 66,000-cubic-foot-per-minute capacity roof fans were installed approximately 40 feet above the furnace at our recommendation following the original test melt on the uranium contaminated scrap. By means of these fans and the manipulation of doors and windows, it was possible to bring the supply air into the building through ground-floor openings at locations affording the maximum dilution to the affected working area.

Following the melt, the 56 tons of steel ingots were stored at the steel mill awaiting a decision as to a suitable method of disposal.

Calculations indicated that the average radium content in the steel ingots on a curie for curie basis compared almost exactly with the uranium found in the ingots resulting from the

uranium contaminated steel. In some instances this was even below that found in steel made by standard methods supposedly using no contaminated scrap. Therefore permission was requested and received from the AEC to sell these ingots. The ingots were individually monitored with a low level beta-gamma survey meter and there was no measurable radiation found.

Film badges worn by the workers during this test melt indicated relatively insignificant exposures to external radiation. The exposures were all less than 50 mrad (millirads) for the two-day period. This was largely due to the fact that the massiveness of the operation was not conducive to manual handling of materials, thus reducing the exposure to external radiation by virtually eliminating direct contact with the metal.

In late 1960 this same smelter received an AEC license to melt uranium contaminated scrap metals. Since that time the uranium contaminated scrap which had accumulated at the FMPC since October, 1958, has been sold outright to this company. At the present time application has been made to the AEC Division of Licensing to amend the license to include the handling of uranium-radium contaminated ferrous metal scrap. If the amendment is granted as was requested, this will then permit the direct sale of uranium-radium contaminated scrap to the licensee. This will greatly simplify the handling of these contaminated materials in the future.

Conclusions

1. Radium in ferrous scrap metal reacts very similarly to uranium in that it is virtually all removed in the slag.

2. The purified metal could cause no health hazard to the steel fabricators or to the general public who might eventually use products manufactured from the steel.

3. Workers can safely carry out the smelting of radium contaminated steel if good general ventilation, such as was described earlier, is provided along with other relatively simple industrial hygiene controls. These should include the wearing of adequate respiratory protection while working with the contaminated scrap or slag or while working in the immediate area of the furnace. Protective clothing should also be worn to minimize the possibility of radium being carried home on the clothing of the workers. Good personal hygiene should be followed to minimize the possibility of ingestion of radium from contamination of the hands and face.

4. Contaminated scrap metals should be mixed with non-contaminated scrap when possible and practical. This will further reduce the personnel exposures and still not contaminate steel to significant levels. In fact, it will further reduce the average concentration in the resultant steel.

Acknowledgments

This study was greatly aided by the complete cooperation extended by the steel mill management. Without their cooperation, the test would have been far less successful and the general results could not have been as comprehensive and conclusive. Their strict enforcement of the wearing of respirators and protective clothing resulted in a minimal exposure of personnel.

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Radiation Alarm Pedestal

WILLIAM H. RAY

*General Atomic, Division of General Dynamics Corporation, P. O. Box 608,
San Diego 12, California*

Ion chamber radiation survey instruments have been equipped with a contact-making meter. They are also equipped with an ordinary electrical receptacle so they may be plugged in to a pedestal when not being used to make a survey. The pedestal may be located to keep the instrument in a convenient position for the user. The pedestal contains a battery-powered alarm which will sound when the radiation dose rate attains the level at which the contact meter is set.

EXCEPT where a source will be at a predetermined locus; such as within process piping or spilled at a specific spot on the floor, remotely recording or remotely reading so called radiation area monitors are of little avail.

It is generally impractical to equip a space with a lattice of detectors sufficient to remotely register the distribution and character of a stray source that may put in its appearance, and scanning or triangulation equipment is likely to cost more than it's worth. As every health physics surveyor knows; a radiation reading without information relating the location of the datum to that of the source or without information regarding the relationship of personnel to the source and the measuring locus is insufficient to permit an appraisal of exposure.

Although automation of the radiological safety survey may be thus dismissed, there is a need wherever the radiation field may change abruptly due to equipment starting, sources being moved, shielding disassembled, etc., to sound a warning to affected personnel of an increase in the dose rate above that which they were prepared to endure. (1) Reactor or accelerator facilities where sources may be generated and withdrawn, (2) operating galleries where radioactive material may back up in control piping, be introduced by malfunctioning ventilation, or be brought in by *Homo sapiens* and other vectors, and (3) ordinary laboratories, where the occupants (or their neighbors) may be manipulating even tracer quantities of radioactive material, all are situations where an increase in the radiation level should be brought to someone's attention so that an appraisal of the radiological hazard could be made promptly

by a survey with suitable instruments operated by a qualified individual.

To provide a radiation alarm without duplicating the detection circuits used in the survey instruments that would already be provided for facilities like those mentioned above, General Atomic has manufactured a pedestal (Figure 1) on which a commercially available radiation survey instrument may repose in operating condition for the convenience of the facility occupants, and which at the same time contains a built in battery powered alarm that will be actuated by any dose rate at which the contact meter of the radiation survey instrument is set.

For an extra \$100 the instrument manufacturer substitutes a contact-making microammeter for the regular meter in his instrument. To match the male plug on the pedestal (Figure 2) we mount a receptacle in the bottom of the instrument case and connect it to the meter contact terminals. Regular 110 volt type plugs and receptacles are used so that any extension cord may be employed to locate the detecting element where it can best serve its alarm function. The meter contacts will never have the power main connected because they are served by a receptacle. 110 volts inadvertently applied to the pedestal plug would be harmless because of the resistors used in each lead.

The alarm may be either a bell or a horn. Horns are used exclusively for radiation alarm purposes at General Atomic. The relay that operates the battery-powered horn has spare contacts which may be connected into other circuits to shut down reactors, actuate a central alarm system, or operate equipment such as valves, doors, fans, etc. The instrument may serve as a criticality alarm.

The versatility of the alarm pedestal is enhanced by utilizing it with a logarithmic scale

Presented at the Twenty-second Annual Meeting of the American Industrial Hygiene Association, Detroit, Michigan, April, 1961.

survey meter with a wide sensitivity range. Our instruments are purchased with two 3-decade ranges, .05 mr/hr to 50 mr/hr, and 50 mr/hr to 50 r/hr.

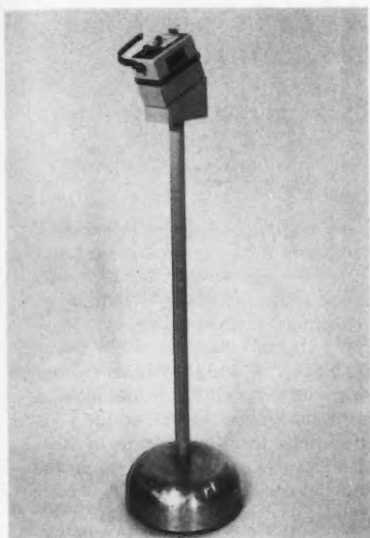


FIGURE 1. Radiation alarm pedestal with detecting instrument in place.

The floating grid electrometer in these instruments requires that measures be taken to minimize the induction of pulses in the electrometer circuit when opening the meter contact circuit. Grid rectification of pulses induced with respect to the capacitance of the ion chamber charge the grid negatively until it is bled away by radiation or by small currents within the electrom-

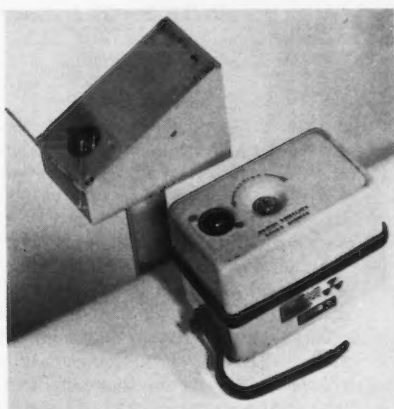


FIGURE 2. Close-up showing standard male electrical plug in pedestal and receptacle mounted in bottom of survey meter.

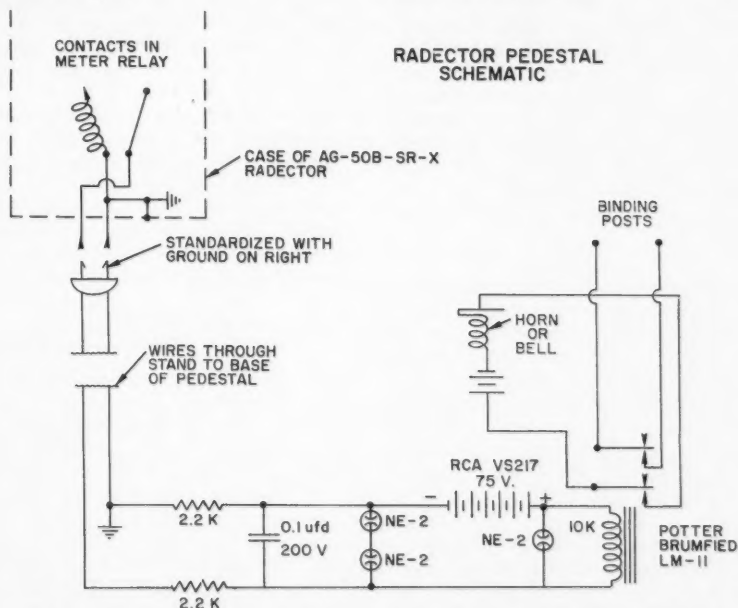


FIGURE 3. Schematic diagram of electrical circuit for radiation alarm pedestal.

eter tube. A pedestal alarm can be silenced only by unplugging the survey instrument to permit the holding coil to release the meter movement from its contact. (Normally one would want to grab the meter and make a survey upon the sounding of an alarm.)

The use of damping circuits, and attention to the polarity of the relay battery have made it possible to unplug the survey instrument without driving the meter movement off the lower end of the scale. The circuit for the pedestal is shown in Figure 3.

PHS DIVISION STAFF

THE DIVISION OF OCCUPATIONAL HEALTH, U. S. Public Health Service, has had a number of additions and changes in its staff during the recent months. In order that our readers may keep abreast of such matters in relation to the principal Federal activity in industrial hygiene, we are briefly mentioning some of these changes. Dr. W. Clark Cooper has been named deputy chief of the Division and will continue to serve as chief of Program Planning and Analysis. Dr. Otto A. Bessey has joined the staff as chief of Research and Training Grants and will be responsible for the direction of the Division's research grants program. Approximately \$1.9 million has been allocated this year for the support of research projects. Three new members have been added to the State Services Branch: William A. McQuary as chief of the State Aid Section; Dr. Gordon S. Siegel as chief of the Employee Health Programs Section; and Miss Mary Louise Brown as chief of the Occupational Nursing Section. Mrs. Mabelle J. Markee is joining Dr. Siegel in the Employee Health Programs Section. Dr. Philip E. Enterline has been assigned to the office of the Division chief and will serve as chief of biometrics and social studies.

In Cincinnati, Ohio, at the Occupational Health Research & Training Facility the newly established Physiology Section has as its chief, Dr. Austin Henschel. Also joining the staff of this new Section are Dr. Alexander Cohen, Dr. Francis Dukes, and Dr. Robert C. Stroud.

Radiation Dose Received by Passengers and Crew on Planes Carrying the Maximum Number of Radiation Units

D. M. DAVIS, J. C. HART, and A. D. WARDEN

Health Physics Division, Oak Ridge National Laboratory, Union Carbide Nuclear Company, Oak Ridge, Tennessee

The dose of ionizing radiation received by passengers and crew members traveling on commercial airlines which transport radioisotopes is of interest to all persons who travel the airways frequently. Tariff regulations permit the air transport of 40 units of radioactive materials where one unit represents a quantity of radioisotopes which when packaged delivers a dose rate of one mr/hr at a distance of one meter from the center of the package. Using five different types of commercial aircraft, shipments of radioisotopes were simulated and dose levels determined at various locations in the aircraft under conditions normal to commercial travel. The survey indicates that the tariff regulations should be amended giving consideration to the type of aircraft involved.

Introduction

OFFICIAL Air Transport Restricted Articles Tariff No. 6C contains the rules and regulations governing the transportation of radioactive materials by air. The regulations which determine the maximum radiation dose which might be received by passengers and crew are given in Appendix I.

Since the regulations set forth in Tariff No. 6C are based on calculations, some direct measurements were made to determine radiation exposures which might occur with the current regulations for comparison with the permissible dose from external sources of ionizing radiation as recommended in *National Bureau of Standards Handbook 59*. Measurements were made on five types of airplanes and results are given in this report.

Equipment and Instrumentation

The planes on which measurements were made were a DC-3, DC-6, DC-7, Convair 340, and a Viscount. (Only one plane of each series was used.) The DC-3 was made available at the Knoxville Airport, and the other planes were made available at the Atlanta Airport.

Readings below approximately 10 mr/hr were made with GM survey type meters, and all measurements above this level were made with the X-10 "slotted chamber" cutie pie. The instruments were calibrated against a standard radium source.

Two sources were used for the measurements. One was Cs^{137} (approximately 162 mc) and the other Co^{60} (approximately 36 mc). The Co^{60} source, as packaged, read 60.0 mr/hr at one meter from the center of the package. The Cs^{137} source read 50.0 mr/hr at one meter. All dose rates in this report are normalized to 40 mr/hr at one meter which is equivalent to 40 "units". (See Rule No. 14 under Appendix I.) Since the sources were packaged in one container rather than in four containers as would be required for shipping (see Item No. 160 Part I (g) Appendix I), the readings represent the maximum for a total shipment of 40 units, regardless of how the individual packages may be arranged.

Procedures

The Co^{60} source was placed in a shipping container, packaged in the same manner as a regular isotope shipment, and placed in the aft cargo compartment of an aircraft. Measurements were made in locations which would be occupied in flight by passengers or crew members. The source was then moved to the other cargo compartments indicated and similar measurements were made. The same procedure was followed for the Cs^{137} source and for all types of planes.

Figure 1 through 4 indicate the types of planes for which measurements were made (except Viscount for which no sketch was available) and the locations at which the sources were placed. While measurements were being made

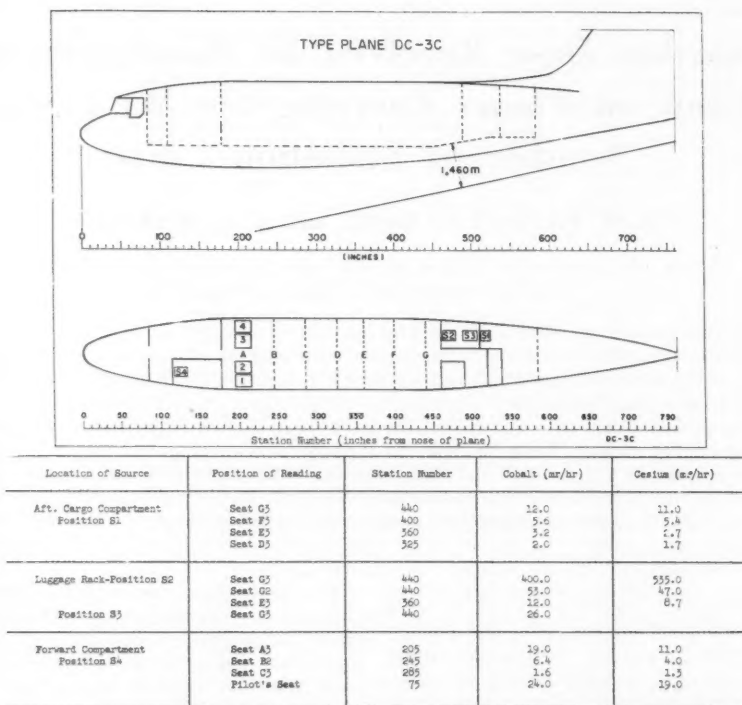


FIGURE 1. The diagrams show the locations of storage spaces (S1, S2, S3 and S4) in the DC-3C plane. The table gives the radiation dose readings at several designated places in the personnel and passenger sections.

with one source, the other source was placed in a lead shield located several feet from the planes.

Data

Table I and the tables at the bottom of Figures 1 through 4 show typical readings from the source when placed in the cargo compartments indicated. All readings have been normalized to 40 units, i.e., the Co^{60} readings were multiplied by 0.67 (ratio of 40 to 60) and the Cs^{137} readings were multiplied by 0.80 (ratio of 40 to 50).

Interpretation of Data

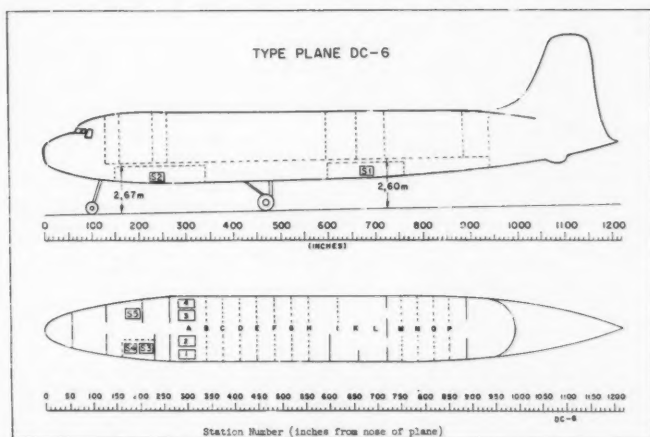
Based on dose rates as recorded in Table I and Figures 1 through 4, it appears that the dose rate for personnel nearest the source can be estimated to a fair degree of accuracy by a simple application of the inverse square law. In these areas of highest dose rate, the attenuation from the shielding materials is not appreciably greater than the build-up from scattering.

There is more shielding interposed between person and source as the distance from the source increases; but distance alone, several meters from the source, is sufficient to reduce the radiation dose rate to an insignificant level.

When isotopes are packaged and shipped ac-

TABLE I
Typical Readings in Viscount

Location of Source	Position of Reading	Station Number	Cobalt (mr/hr)	Cesium (mr/hr)
Fwd. cargo compartment	Seat 1	Drawing for Viscount not available	20.0	13.0
	Seat 2		16.0	13.0
	Seat 3		8.0	6.7
	Seat 4		5.6	5.4
Aft. cargo compartment	Seat 1		13.0	
	Seat 2		5.6	
	Seat 3		3.2	
	Seat 4		1.6	



Location of Source	Position of Reading	Station Number	Cobalt (mr/hr)	Cesium (mr/hr)
Aft. Belly Compartment Position S1	Seat I5	680	7.2	5.0
	Seat K3	655	24.0	15.0
	Seat L3	700	16.0	12.0
	Seat M	750	8.0	6.7
Fwd. Belly Compartment Position S2	Seat A3	505	13.0	
	Seat B3	540	6.4	
	Seat C3	575	3.2	
	Seat D3	410	1.6	
Fwd. Upper Compartment Position S3	Men's Lounge	238	48.0	
	Seat A5	505	16.0	
	Seat B5	540	9.6	
Fwd. Upper Compartment Position S4	Pilot	110	8.0	
	Co-Pilot	110	8.0	
	Radio Operator	270	16.0	
Luggage Compartment Position S5	Radio Operator	170	64.0	
	Navigator	152	24.0	

FIGURE 2. The diagrams show the locations of storage spaces (S1, S2, S3, S4 and S5) in the DC-6 plane. The table gives the radiation dose readings at several designated places in the personnel and passenger sections.

cording to current regulations, particularly with respect to minimum distance to crew members and passengers, no crew member or passenger will receive a significant exposure as interpreted from *NBS Handbook 59*. The maximum exposure would not exceed 10 mr/hr if all regulations are enforced.

Discussion

The regulations which determine the rate of exposure from isotope shipments are stated briefly as follow:

- (1) Maximum of 40 units per shipment.
- (2) Forty units must be a minimum of 6 feet from nearest personnel. (This is 6 feet to nearest package.)
- (3) Limit of 10 units per package.
- (4) Maximum of 200 mr/hr at surface of package.

For isotopes of penetrating gamma radiation, the size of the package for maximum exposure rates would be 44.72 cm in diameter. (Reading at 1 m = 10 mr/hr $\therefore 100^2/X^2 = 200/10$.) At 6 feet from the surface of this package, the reading would be approximately 2.3 mr/hr (6 feet + 22.36 cm), Figure 5. Assuming four packages of this type, ten units per package, the total reading would be approximately 9.2 mr/hr (2.3×4).

To indicate the maximum exposure a person might receive, the following hypothetical case will serve as an example. Take the case of a person who (a) averages one flight per week, (b) on planes carrying isotopes, (c) in which he is the person nearest to the source, and (d) the average flight is approximately three hours; then the maximum probable exposure would not exceed 30 mr/wk ($9.2 \times 3 = 27.6$ mr).

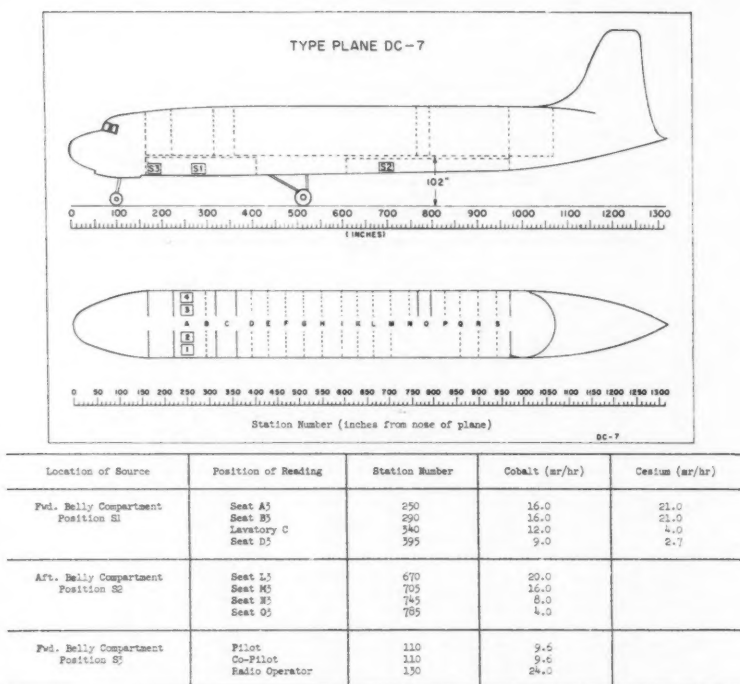


FIGURE 3. The diagrams show the locations of storage spaces (S1, S2 and S3) in the DC-7 plane. The table gives the radiation dose readings at several designated places in the personnel and passenger sections.

(This does not differ appreciably from the value calculated by Robley D. Evans in *Nuclear Science Series, Preliminary Report No. 11.*)

When the regulations are checked against the cargo spaces available in the various planes, it is evident that some types of planes cannot carry the maximum of 40 units of penetrating gamma emitting isotopes. For example, the main floor of the DC-6, Figure 2, is 92 cm above the floor of the cargo compartment. (There are two cargo compartments on the DC-6. Both are under the main floor.) The regulations state that packages of 10 units (10 mr/hr at one meter) must be a minimum of 3 feet from the nearest person. If we add the diameter of the container suggested above for maximum exposure to the 3 feet required by regulations, the distance from the floor of the cargo space to the main floor would have to be 136.17 cm.

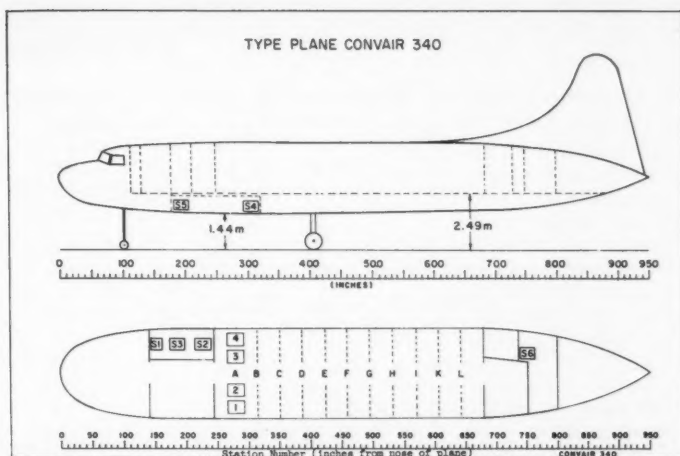
The data in Table I and Figures 1 through 4 emphasize also the fact that exposures in excess of 10 mr/hr (the approximate maximum value when all regulations are enforced) may result when 40 units of Co^{60} or Cs^{137} are carried on

some types of planes. Unless due caution is taken in loading the isotope in the proper cargo compartment, exposures well above 10 mr/hr may be encountered.

Suggestions

Studies should be made to determine what changes in cargo space are necessary to permit airplanes to carry the maximum amount of isotopes (40 units) without violating one or more of the regulations. (According to literal interpretation of current regulations, some planes should not carry over 5 units of gamma emitting isotopes such as Co^{60} .) For example, it might be feasible to provide isotope shipment space in the wing, the wheel well, or a remote section of the fuselage.

The "Table for Personnel Separation" (see Appendix I) should be extended to the maximum distance obtainable in the various types of planes and the "40 unit maximum" dropped from the regulations. The "number of units" vs "distance in feet" could be given in the form of a graph as illustrated in Figure 6.



Location of Source	Position of Reading	Station Number	Cobalt (mr/hr)	Cesium (mr/hr)
Fwd. Cargo Compartment Position S1	Flight Engineer	120	78.0	
	Pilot	85	16.0	
	Co-Pilot	85	16.0	
Fwd. Cargo Compartment Position S2	Seat A)	275	114.0	67.0
	Seat B)	310	20.0	15.0
	Seat C)	350	9.5	6.7
	Seat D)	385	6.4	4.0
Fwd. Cargo Compartment Position S3	Co-Pilot	85	8.0	
	Flight Engineer	120	19.0	
	Seat A)	275	24.0	
Lower Cargo Compartment Position S4	Underneath Plane(contact)	310	800.0	
	Seat A)	275	20.0	
	Seat C)	350	46.0	
Lower Cargo Compartment Position S5	Flight Engineer	120	16.0	
	Co-Pilot	85	8.0	
	Seat A)	275	34.0	
Aft. Cargo Compartment Position S6	Seat L)	640	18.0	
	Seat K)	605	5.5	
	Seat J)	570	2.4	
	Seat H)	530	1.6	

FIGURE 4. The diagrams show the locations of storage spaces (S1, S2, S3, S4, and S5) in the Convair 340 plane. The table gives the radiation dose readings at several designated places in the personnel and passenger sections.

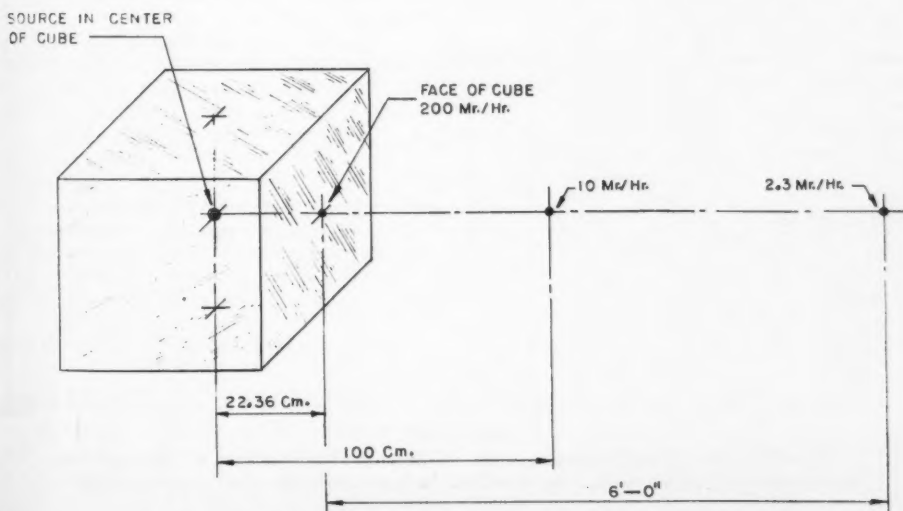


FIGURE 5. The size of package for the maximum exposure rate.

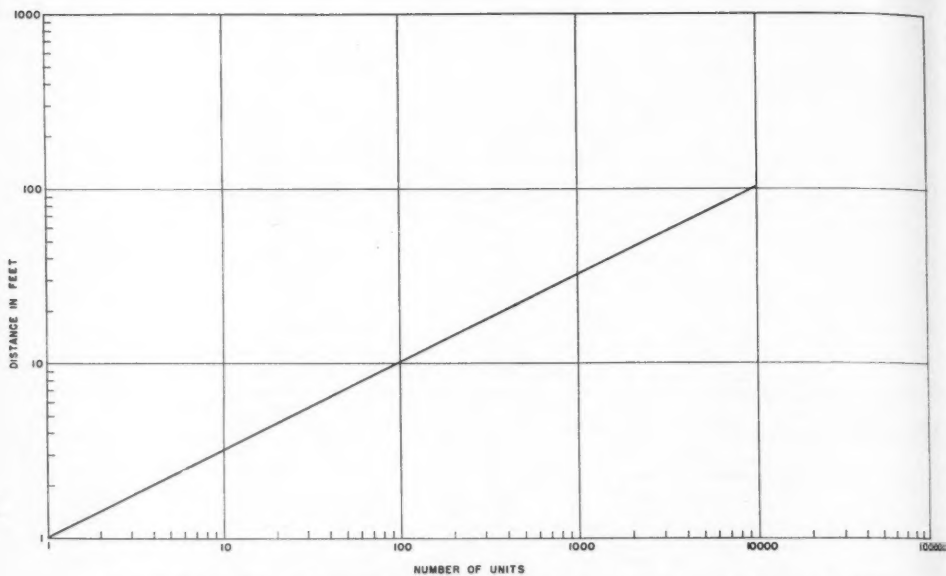


FIGURE 6. Graphical representation of the relationship of the distance of personnel from the radiation source and the number of units for isotope shipments.

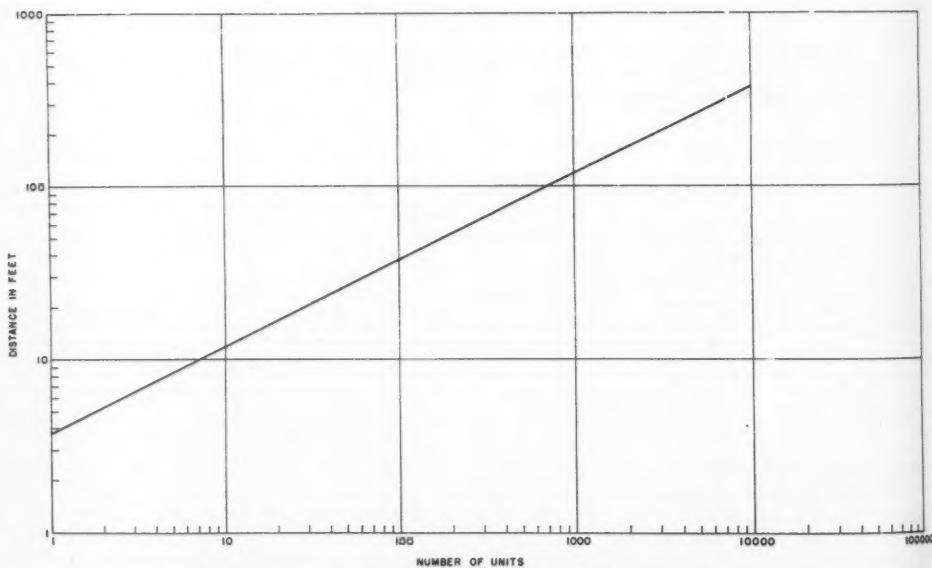


FIGURE 7. Graphical representation of the relationship of the distance of personnel from the radiation source and the number of units of isotopes for storage at an airport terminal.

A "Table for Personnel Separation" or perhaps a graph as suggested above should be prepared for isotope storage at the terminals since there is no provision in the regulation for storage prior to shipment. Figure 7 is an illustration of a relationship of units to distance that would limit the exposure to 30 mr/wk, assuming an eight-hour work day and a five-day work week.

With these exceptions, the regulations as stated, as far as exposure rates are concerned, seem to be in order.

Appendix I

Excerpts from *Official Air Transport Restricted Articles Tariff No. 6C*

Section 1 Rule No. 6 (2nd revision page 10)

4. **Class D—Radioactive Materials:** A radioactive material is any material or combination of materials which spontaneously emits ionizing radiation. For the purpose of these rules radioactive materials are divided into three groups according to the type of radiation emitted at any time during transportation, as follows:

Group I—radioactive materials: Are those materials which emit any gamma radiation, either alone or with electrically charged particles or corpuscles.

Group II—radioactive materials: Are those materials which emit neutrons and either or both of the types of radiation characteristic of Group I radioactive materials.

Group III—radioactive materials: Are those materials that emit electrically charged corpuscular rays only, i.e., alpha or beta, etc., or any other that is so shielded that the gamma radiation at the surface of the package does not exceed 10 milliroentgens for 24 hours at any time during transportation.

- (r) **Unit of (gamma radiation):** Is one milliroentgen per hour at a meter for "hard gamma" radiation; i.e., that amount of gamma radiation which will have the same effect on sensitive photographic film as one milliroentgen per hour at a meter of "hard gamma" radiation of radium filtered through $\frac{1}{2}$ (0.5, 1.27 cm) inch of lead.

Section I Rule No. 14 (1st revision page 12)

- (b) Not more than 40 units of radioactive material Group I or II will be carried on any aircraft.

Section IV (Item No. 16e)

Part I. (See T. C. George's Tariff No. 13, Part 73.393 (3) and (g), ICC Regulations for Rail Express.)

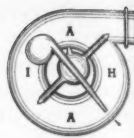
- (e) All outside shipping containers must be of such design that the gamma radiation will not exceed 200 milliroentgens per hour or equivalent at any point of readily accessible surface. Containers must be equipped with handles and protective devices, when necessary in order to satisfy this requirement.
- (g) Radioactive materials Group I, liquid, solid, or gaseous, must be packed in suitable inside containers completely surrounded by a shield of lead or other suitable material of such thickness that at any time during transportation the gamma radiation at one meter (39.3 inches) from any point on the radioactive source will not exceed 10 milliroentgens per hour. The shield must be so designed that it will not open or break under conditions incident to transportation. The minimum shielding must be sufficient to prevent the escape of any primary corpuscular radiation to the exterior of the outside shipping container.

Part II. Handling (in Aircraft)

- (a) (Applicable only to Groups I and II radioactive materials.) A container or group of containers of Group I or II radioactive materials shall not be placed closer than the distance specified in the following table to any area that may be continuously occupied by crew members or passengers. If more than one such container is present, the distance shall be computed from the table by adding together the number of units shown on the label of each package.

Total Number of Units	Minimum Distance to Crew Members and Passengers
0-2.....	1 foot
3-5.....	2 feet
6-10.....	3 feet
11-20.....	4 feet
21-30.....	5 feet
31-40.....	6 feet

Distance means the number of feet from the nearest edge of the nearest radioactive container.



HYGIENIC GUIDE SERIES

Benzene

(Benzol)

(Revised 1961)

I. Hygienic Standards

- A. RECOMMENDED MAXIMUM ATMOSPHERIC CONCENTRATION (8 hours): 25 parts of vapor per million parts of air, by volume (ppm).¹

1. *Basis for Recommendation:* Principally human experience in industry plus toxicological observations on animals.

B. SEVERITY OF HAZARDS:

1. *Health:* Moderate for acute exposure; high for chronic. Absorption occurs chiefly by inhalation. High concentrations irritate the respiratory tract and produce narcosis. Repeated exposure to benzene may cause bone marrow damage, resulting in a decrease in the circulating white blood cells, platelets, and red blood cells. The red cells may occasionally show an increase in size in early poisoning.⁷ Many serious illnesses and fatalities have occurred in association with chronic exposures to benzene. It has been stated that symptoms may occasionally occur after exposure has ceased.⁵ Individual susceptibility varies widely with the remote possibility that an occasional individual may be affected by prolonged exposure to 25 ppm. A primary irritant type of dermatitis may result from repeated skin contact. Percutaneous absorption is considered insignificant. Gerarde^{3, 4, 5} has shown that alkyl derivatives do not produce the bone marrow effects of benzene in rats.

2. *Fire:* High. Explosive limits are 1.4–7.1% by volume at 212°F. Flash point is –11.1°C (12.0°F) (closed cup).

- C. SHORT EXPOSURE TOLERANCE: For man on single exposure, 3000 ppm is endurable

for 30 to 60 minutes; 7500 ppm is dangerous in 30 to 60 minutes.¹⁰

- D. ATMOSPHERIC CONCENTRATION IMMEDIATELY HAZARDOUS TO LIFE: 20,000 ppm is reported fatal in 5 to 10 minutes.¹⁰

II. Significant Properties

Benzene is a flammable, colorless, odorless liquid

Chemical formula:	C ₆ H ₆
Molecular weight:	78.11
Specific gravity:	0.8790 (20°/4°C)
Boiling point:	80.1°C (760 mm Hg)
Vapor pressure:	95.14 mm Hg at 25°C
Solubility:	In most organic solvents

At 25°C and 760 mm

Hg:

1 ppm of vapor:	0.0032 mg/liter
1 mg/liter of vapor:	313 ppm
Saturated air concentration:	125,000 ppm
Relative density of saturated air:	1.21 (air = 1)
Relative density of vapor:	2.7 (air = 1)

Note: Benzene, an aromatic hydrocarbon, is to be distinguished carefully from benzine, a petroleum distillate containing mixed hydrocarbons (such as pentane and hexane) in uncertain proportions. However, some commercial petroleum and aromatic solvents may contain quantities of benzene of possible hygienic significance. In the past, some gasolines contained appreciable quantities of benzene, but currently the amount does not exceed 3%. An accurate estimation of the quantities of benzene present in such solvents is valuable in estimating the benzene hazard.

III. Industrial Hygiene Practice

- A. RECOGNITION: Benzene may be recognized by its characteristic odor at concen-

The Committee wishes to acknowledge the assistance of Dr. L. J. Goldwater and Dr. H. W. Gerarde in the preparation of this Hygienic Guide.

trations of about 100 ppm.¹⁰ It is an excellent solvent for a wide variety of gums, resins, fats, alkaloids, and rubber and is frequently used in combination with other solvents. It is present, to some extent, in most gasolines and is a common ingredient of paint and varnish removers.

B. EVALUATION OF EXPOSURES:

1. *Instrumentation:* Direct determination may be made by the commercially available aromatic hydrocarbon detecting instruments, if the sensitivity is 25 ppm or lower. It may also be determined by scrubbing the air through spectroscopically pure isooctane or alcohol in an all-glass device, followed by ultraviolet spectrophotometry, or by adsorption on silica gel, with subsequent extraction and ultraviolet spectrophotometry.⁸

2. *Chemical Method:* Collection by adsorption on silica gel, followed by nitration, color development with sodium hydroxide and comparison with prepared standards,² or by polarography.⁶ Detector tubes are available.

C. RECOMMENDED CONTROL PROCEDURES:

Maintain workroom atmospheres below 25 ppm by means of process enclosure and/or ventilation. Substitute, wherever possible, a less toxic solvent. Prevent skin contact through the use of protective clothing made of nitrile type rubber (Butaprene, Chemigum, Hycar, Krynac, Paracril, Tylor) or Neoprene. Use chemical type goggles.

IV. Specific Procedures

A. *FIRST AID:* Remove from exposure; remove contaminated clothing, flush eyes with water, and wash contaminated skin areas with soap and water.

B. SPECIFIC MEDICAL PROCEDURES:

1. *Preplacement:* This should include a complete blood count as well as a careful history and physical examination. Particular attention should be paid to a history of any type of blood disturbance; but any chronic disturbances of general health, particularly those which might involve liver or renal diseases, should be carefully considered by the physician.

2. *Periodic Examination:* A program of routine physical examinations is essential for all persons regularly exposed to benzene and especially if exposures

are at or possibly above the threshold limit. This should include a complete blood count at intervals no greater than every two months (frequency may be decreased if regular air analyses show the continued absence of hygienically significant amounts of benzene).

The measurement of urinary sulfates has been suggested as a control procedure.¹³ The determination of urinary phenols has also been suggested as an index of personnel exposure, particularly useful where marked fluctuations in atmospheric concentrations occur or where the operations are intermittent or varied.¹² Values above 200 mg of total phenol per liter of urine on spot samples collected late in the day are stated to be significant.⁹ While both of these methods are measures of benzene exposure, they tell nothing of the health of the individual, but may be of some use as control procedures, particularly in low-grade chronic exposures. They are of most value when obtained from groups of exposed personnel, rather than from single individuals.

V. References

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Bromochloromethane

(Methylene Chlorobromide, BCM)

I. Hygienic Standards

A. RECOMMENDED MAXIMUM ATMOSPHERIC CONCENTRATION (8 hours): 200 parts of vapor per million parts of air, by volume has been "tentatively" recommended.¹

1. *Basis for Recommendation*: Primarily on results of repeated administration to animals.^{2, 4, 5}

B. SEVERITY OF HAZARDS:

1. *Health*: Probably moderate, for both acute and chronic exposure. Knowledge of effects in humans is limited at present. Anesthesia is the primary hazard from single exposure to high concentrations and organic (liver) injury is likely only if severe anesthetic effects are apparent. Repeated exposure to moderately excessive concentrations is likely to result in slight liver injury. The presence of increased blood inorganic bromide appears to indicate some hydrolysis *in vivo*.² The material has a low oral toxicity and does not present a problem from skin absorption. Bromochloromethane is slightly irritating to the eyes and has a defatting effect on the skin.

2. *Fire and Explosion*: None. This material is used as a fire extinguishing fluid. Pyrolysis products may be hazardous.

C. SHORT EXPOSURE TOLERANCE: Little or no injury was seen in animals from single exposure to 2500 ppm for one hour, or to 900 ppm for four hours. Death from anesthesia occurred in animals when exposures exceeded 4% (40,000 ppm) for six minutes or 2% (20,000 ppm) for 15 minutes.⁵

D. ATMOSPHERIC CONCENTRATION IMMEDIATELY HAZARDOUS TO LIFE: Not known, but probably 2-4%. Animal experiments indicate that if exposure is terminated before death occurs, complete and rapid recovery is likely.⁵

II. Significant Properties

Colorless liquid with a typical "chlorinated hydrocarbon" odor:

Chemical formula:	CH ₂ BrCl
Molecular weight:	129.40
Specific gravity:	1.93439 (20°/4°C)
Boiling point:	68.11°C at 760 mm Hg
Vapor pressure:	147.17 mm Hg at 25°C
Relative density at 25°C:	4.47 (dry air = 1.0)
Relative density of saturated air at 25°C:	1.67 (dry air = 1.0)
Air saturated at 25°C and 760 mm Hg contains:	194,000 ppm
Solubility:	infinite in acetone, carbon tetrachloride, benzene, n-heptane and ethanol
At 25°C and 760 mm Hg:	
1 ppm vapor:	0.005294 mg/liter
1 mg/liter:	188.9 ppm

III. Industrial Hygiene Practice

A. RECOGNITION: Bromochloromethane is used as a fire extinguishing fluid. It is also used to some extent as a solvent and as a refrigerant.

B. EVALUATION OF EXPOSURES:

1. *Instrumentation*: Instruments based on the Beilstein test such as the Davis Halide Meter may be calibrated for bromochloromethane. Instruments employing measurement of the conductivity of water which has absorbed the combustion products of this material are available, and those employing hy-

drogen flame ionization, while almost completely nonspecific, can probably be used. Infrared and mass spectrographic methods can be used, especially when absolute identification is necessary.

2. **Chemical Analysis:** Direct combustion followed by absorption in a basic reducing solution and then determination of the halide ion has been used successfully, as has adsorption on silica gel followed by thermal desorption and combustion.³ Halogenated hydrocarbon "detector tubes" by various manufacturers can probably be calibrated for bromochloromethane.

- C. **RECOMMENDED CONTROL PROCEDURES:** Fire extinguishers containing bromochloromethane should only be used on outdoor or other fires in areas of good ventilation. If used in small, confined or poorly ventilated locations, the area should be evacuated immediately. Since pyrolysis products of bromochloromethane, like other halogenated hydrocarbons, are more toxic than the compound itself, these areas should be ventilated thoroughly before they are re-entered.⁶ Time-weighted average concentrations to which men are chronically exposed should not exceed 200 ppm and "peak" exposures should not exceed 400 ppm. Gross skin contact should be prevented; suitable eye protection should be provided.

Carbon Tetrachloride

(Revised 1961)

I. Hygienic Standards

- A. **RECOMMENDED ATMOSPHERIC CONCENTRATION** (8 hours): 25 parts of vapor per million parts of air, by volume (ppm).¹ It is felt that this is a maximum which should not be exceeded for repeated daily exposures. The time weighted average should not exceed 10 ppm.² **These levels cannot be detected by odor.**
 1. **Basis for Recommendation:** Human experience and observation of experimental animals.³

IV. Specific Procedures

- A. **FIRST AID:** Remove person to uncontaminated atmosphere; use artificial respiration and oxygen if indicated. Remove wet clothing and do not allow it to be re-worn until thoroughly dry. If eyes are contaminated, they should be flushed with large amounts of water for 15 minutes and medical attention obtained.
- B. **SPECIFIC MEDICAL PROCEDURES:** Placement examinations should consider any previous liver disease. In case of repeated exposures, tests of hepatic function and possibly blood bromides might be useful. Epinephrine should not be used in cases of anesthesia with BCM because of the possibility of ventricular fibrillation.

V. References

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2. Highman, B., J. L. Svirbely, W. F. von Oettingen, N. C. Alford and L. J. Pecora: *Arch. Pathol.* 45: 299 (1948).
3. Peterson, J. E., H. F. Hoyle, and E. J. Schneider: *Amer. Ind. Hyg. Assoc. Quart.* 17: 429 (1956).
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B. SEVERITY OF HAZARDS:

1. **Health:** High for both acute and chronic exposures. Narcosis with subsequent liver and kidney injury and death may occur as the result of an acute overexposure. An acute nephrosis and anuria may occur from single exposures which are insufficient to cause symptoms of narcosis or of irritation. Chronic inhalation exposure to concentrations above 25 ppm may also result in severe injury to the kidneys and liver.³ Persons who have consumed

alcohol are more susceptible to such effects than others, but individual susceptibility varies widely. Prolonged or repeated contact with the liquid may result in irritation of the skin. Liquid carbon tetrachloride can be absorbed through the intact skin of experimental animals in toxic amounts.⁵ Eye contact results in pain and minimal injury to the conjunctiva. It is highly toxic if ingested.

2. *Fire*: None. Carbon tetrachloride is used as a fire extinguishing agent. Thermal decomposition products are quite toxic.
- C. **SHORT EXPOSURE TOLERANCE**: Animal experiments showed little or no injury from single exposures to 300 ppm for one hour, 90 ppm for four hours, or 2000 ppm for six minutes.³
- D. **ATMOSPHERIC CONCENTRATION IMMEDIATELY HAZARDOUS TO LIFE**: Unknown, but probably about 2%. Human fatalities from acute renal damage have occurred after one-half to one hour exposure to concentrations of 1000–2000 ppm.⁴

II. Significant Properties

Carbon tetrachloride is a volatile, non-flammable, colorless liquid with a sweetish, aromatic odor. The odor threshold is about 50 ppm of vapor.⁵ Odor is usually not objectionable at acutely toxic levels and may not even be apparent at concentrations which are harmful upon repeated exposure.

Chemical formula: CCl_4
 Molecular weight: 153.84
 Specific gravity: 1.5843 (25°/4°C)
 Boiling point: 76.54°C at 760 mm

Hg
 Vapor pressure at 25°C: 115.25 mm Hg

Solubility: Insoluble in water, but miscible with acetone, alcohol, ether, and most organic solvents.

At 25°C and 760 mm of Hg:
 1 ppm of vapor: 0.00628 mg/liter
 1 mg/liter of vapor: 159 ppm
 Saturated air concentration: 15.2%
 Relative density of vapor: 5.3 (air = 1.0)
 Relative density of saturated air: 1.65 (air = 1.0)

III. Industrial Hygiene Practice

- A. **RECOGNITION**: Carbon tetrachloride is used as a degreasing and cleaning agent, as a fire extinguisher, as a solvent in rubberizing fabrics, as a component in fumigant mixtures used in the grain industry, and as a solvent in the chemical processing and manufacturing industries. It is sometimes used as a component of proprietary solvent preparations to reduce their fire hazards. At elevated temperatures its vapors decompose to produce chlorine, hydrogen chloride, and (in presence of ozone, during welding etc.) possibly some phosgene.^{6, 7}

B. EVALUATION OF EXPOSURES

1. *Instrumentation*: Instruments based on the Beilstein test such as leak detectors and the Davis Halide meter may be calibrated for carbon tetrachloride. Instruments employing measurement of the conductivity of water which has absorbed the combustion products of this material are available. Infrared, mass spectrographic and gas chromatographic methods can be used, especially when absolute identification is necessary. Infrared and conductivity instruments are especially suitable for continuous air analysis.

2. *Chemical Analysis*: Direct combustion followed by absorption in a basic, reducing solution and then determination of the halide ion has been used successfully,⁸ as has adsorption on silica gel followed by thermal desorption and decomposition.⁹ Removal from silica gel by isopropanol followed by alkaline hydrolysis can also be used.¹⁰ Halogenated hydrocarbon "detector tubes" by various manufacturers can probably be used, but each batch should be separately calibrated. A modified Fujiwara reaction may be used.¹¹ None of these methods are specific for carbon tetrachloride.

- C. **RECOMMENDED CONTROL PROCEDURES**: All employees should be instructed in the hazards and control measures. The hazard of severe renal and liver injury may be greatly reduced by substituting a less toxic solvent such as methylene chloride, trichloroethylene, perchloroethylene, or 1,1,1-trichloroethane (methyl chloroform). Petroleum solvents with flash points above 100°F may be useful in some instances as a replacement. Where

carbon tetrachloride is used, local exhaust ventilation will usually be necessary, and frequently the operation should be isolated (by total enclosure) from the general work space. Concentrations to which men are repeatedly exposed should rarely or never exceed 25 ppm and should not average over 10 ppm. Air sampling is mandatory, and an automatic air monitoring system may be necessary to assure adequate control of vapor. Adequate respiratory protection should be provided for maintenance workers or in the event of spills or leaks. Gross skin contact should be prevented. Spectacles will usually offer adequate eye protection.

IV. Specific Procedures

- A. **FIRST AID:** Remove person to uncontaminated atmosphere and apply artificial respiration if indicated. Call a physician at once. Remove wet clothing and cleanse the skin with water or soap and water. Do not allow contaminated clothing to be reworn until it is thoroughly dry. Eye splashes should be treated with copious water irrigation.

Any person sustaining an accidental high level exposure should be observed by a physician for several days, even if no symptoms have been noticed at the time of exposure. Nausea and vomiting occurring twenty-four hours or so after exposure may be the first sign of renal damage.

- B. **SPECIFIC MEDICAL PROCEDURES:** Employment should be restricted to persons free of a history of liver or kidney disorder, or of alcoholism. Treatment of acutely affected individuals should be directed toward restoring kidney and liver function. Hospitalization is essential. Oxygen therapy should be instituted if any anes-

thesia or cardiac failure has occurred. As with other chlorinated hydrocarbons, the use of epinephrine during any anesthetic phase may cause ventricular fibrillation. As the most serious sequelae of acute carbon tetrachloride exposure is toxic nephrosis (and death is directly related thereto), the use of an artificial kidney is indicated and may prove life-saving. Some indication of the extent of exposure may be obtained from an analysis of exhaled air.¹²

V. References

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1, 1, 1-Trichloroethane (Methyl Chloroform)

(Revised 1961)

I. Hygienic Standards

- A. **RECOMMENDED MAXIMUM ATMOSPHERIC CONCENTRATION** (8 hours): 500 parts of vapor per million parts of air, by volume (ppm)¹

1. *Basis for Recommendation:* Repeated

administration to animals and human experience.^{2, 5}

B. SEVERITY OF HAZARDS:

1. *Health:* Moderate for acute exposure, but chronic exposures to concentrations that are without acute effects

are unlikely to produce injury. 1,1,1-Trichloroethane is readily absorbed and excreted through the lungs.³ In acute exposure the most important toxic action is a functional depression of the central nervous system leading ultimately to respiratory failure. Experiments with dogs have indicated that this material can induce ventricular arrhythmia similar to that produced by other chlorinated solvents.⁴ Animals exposed repeatedly to high concentrations exhibited some reversible changes in liver histology, but they were not the severe effects associated with carbon tetrachloride. Chronic exposures to 1000 ppm resulted in moderate fatty degeneration of the liver but no liver necrosis or kidney injury. Growth depression occurred at 650 ppm. No effects were seen from repeated 7-hour daily exposures to 500 ppm. In controlled human exposures to 500 ppm no effects other than slight, transient eye irritation were noted, but at 1000 ppm and above, mild eye irritation was experienced by all subjects, and some became dizzy.^{2, 5} As with most solvents, dermatitis might result from repeated skin contact, but 1,1,1-trichloroethane is only poorly absorbed through the skin. Eye contact may result in pain and discomfort, but no impairment of vision is likely.

2. *Fire*: 1,1,1-Trichloroethane has no flash point when tested by standard ASTM procedures for the TAG Closed or Cleveland Open Cup tests. Some inhibited 1,1,1-trichloroethanes have been found to have explosive limits of 10% to 15.5% in air, but a high energy source is required to cause ignition and, if the ignition source is removed, the vapor will not continue to burn.² When inhibited products are evaporated to a small volume the residue may flash. Thermal decomposition products are very irritating and may be quite toxic, but voluntary overexposure to such materials (largely hydrogen chloride)⁶ is not likely.

- C. *SHORT EXPOSURE TOLERANCE*: Beginning anesthetic effects, including incoordination, appear in some human subjects exposed to concentrations of 800 to 1000 ppm. They occur quickly in humans at concentrations of 2000 ppm or higher.² Exposures to concentrations in excess of

30,000 (3% by volume) may be lethal in 5-6 minutes.⁷

- D. *ATMOSPHERIC CONCENTRATION IMMEDIATELY HAZARDOUS TO LIFE*: 30,000 ppm (3% by volume) based on animal experiments.^{5, 7}

II. Significant Properties

Methyl chloroform is a volatile, colorless liquid with an aromatic "chloroform-like" odor. The odor threshold may range from 20-100 ppm.⁸

Chemical formula:	CH ₃ CCl ₃
Molecular weight:	133.42
Specific gravity:	1.3366 (25°/4°C)
Boiling point:	74.07°C at 760 mm Hg
Vapor pressure at 25°C:	125 mm Hg
Solubility:	Insoluble in water, but miscible with alcohol, ether and most organic solvents.

At 25°C and 760 mm Hg:

1 ppm of vapor:	0.00545 mg/liter
1 mg/liter of vapor:	183 ppm
Saturated air concentration:	16.5%
Relative density of vapor:	4.6 (air = 1.0)
Relative density of saturated air:	1.6 (air = 1.0)

III. Industrial Hygiene Practice

- A. *RECOGNITION*: Methyl chloroform is used as a solvent and as a degreasing and cold-cleaning agent. In many instances it has been substituted for the more toxic carbon tetrachloride.

- B. *EVALUATION OF EXPOSURE*:

1. *Instrumentation*: Instruments based on the Beilstein test such as the Davis Halide Meter⁹ and halide leak detectors may be calibrated for 1,1,1-trichloroethane. Instruments employing measurement of the conductivity of water which has absorbed the combustion products of this material are available, and those using hydrogen flame ionization, while almost completely non-specific, can probably be used. Gas chromatographic, infrared, and mass spectrographic methods can be used, especially when absolute identification is necessary.

2 **Chemical Analysis:** Direct combustion followed by absorption in a basic, reducing solution and determination of the halide ion has been used successfully,¹⁰ as has adsorption on silica gel followed by thermal desorption and combustion.¹¹ The method of Fahy¹² using isopropyl alcohol to desorb from silica gel, followed by alkaline hydrolysis, is said to be applicable.¹⁴ A modified Fujiwara reaction may be used.¹³ Detector tubes by various manufacturers can probably be used, but each batch should be separately calibrated before use.

C. **RECOMMENDED CONTROL PROCEDURES:** Concentrations to which men are repeatedly exposed should not exceed 500 ppm. General ventilation is usually sufficient to maintain such control at room temperature unless large surfaces are wet with the solvent. Halogenated solvents should not be used where their vapors (in concentrations of a few ppm or more) will come into contact with very hot surfaces (such as near a welding operation) because toxic decomposition products such as chlorine and hydrogen chloride may be formed. In such cases local exhaust ventilation will be necessary.

Non-inhibited methyl chloroform should not be used in contact with aluminum because of its reactivity with this metal.

Gross skin contact should be prevented; spectacles will usually offer adequate eye protection.

IV. Specific Procedures

A. **FIRST AID:** Remove victim to an uncontaminated atmosphere and apply artificial respiration if breathing has stopped. Remove wet clothing and do not allow it to be re-worn until it is thoroughly dry. If eyes are contaminated they should be flushed with large amounts of water.

B. **SPECIFIC MEDICAL PROCEDURES:** Treat symptomatically, watch the cardiac rhythm in any case of anesthesia produced by 1,1,1-trichloroethane. Oxygen therapy may be used, but epinephrine is contraindicated. Some indication of the extent of exposure may be obtained from an analysis of exhaled air.⁵ Urinary urobilinogen may be of interest as a liver function test following acute excessive exposures.⁵ Recovery from a non-fatal acute episode can be expected to be complete and prompt.

V. References

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Epichlorohydrin

(1-Chloro-2,3-epoxypropane)

I. Hygienic Standards

A. **RECOMMENDED MAXIMUM ATMOSPHERIC CONCENTRATION (8 hours):** No official

value has been published. Five parts of vapor per million parts of air (ppm) by volume has been suggested on the basis of extensive animal experiments.^{2,3} **Such a level cannot be detected by odor.**

B. **SEVERITY OF HAZARDS:**

The Committee wishes to acknowledge the assistance of U. C. Pozzani in the preparation of this Hygienic Guide.

1. **Health:** High for both acute and chronic exposures of any type. The vapor is highly irritating to the mucous membranes of the eyes and respiratory tract.⁴ Pulmonary edema and renal damage may occur. Deaths occurred among guinea pigs, rats, and rabbits inhaling 500 ppm of vapor for four hours. A few deaths occurred in a group of rats inhaling 62 ppm seven hours per day for 45 days. Some evidence of bronchial irritation was seen in dogs and monkeys after inhaling 16 ppm seven hours per day for 90 days. Studies on rats and rabbits show that epichlorohydrin is highly toxic when administered by mouth or by 24-hour covered application to the skin.³

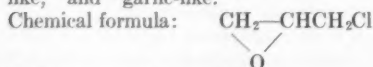
Liquid epichlorohydrin is reported to produce only a slight transient burning sensation on first contact with the skin of man. Blistering and deep-seated pain in the contact area may occur several hours after the time of the initial contact. The liquid is also highly irritating to the eyes.⁴

An occasional case of sensitization with resulting intolerance to very small quantities of epichlorohydrin is reported.⁴

2. **Fire and Explosion:** Flash point 105°F (Tagliabue open cup). The epoxy group readily reacts *exothermically* with alcohols, phenols, carboxy acids, amines, etc.
- C. **SHORT EXPOSURE TOLERANCE:** Six species of laboratory animals tolerated a 4-hour inhalation of 250 ppm but only mice survived 500 ppm with no deaths. Inhalation by humans of 40 ppm during air sampling operations at the site of a spill caused immediate eye, nose and throat irritation but no permanent ill effects.⁵
- D. **ATMOSPHERIC CONCENTRATION IMMEDIATELY HAZARDOUS TO LIFE:** Not known for humans, but 23,000 ppm killed rats in ten minutes but not in five minutes.³

II. Significant Properties

Colorless liquid with distinctive odor described variously as "ethereal," "chloroform-like," and "garlic-like."



Molecular weight: 92.53

Specific gravity:	1.1761 (20°/20°C)
Boiling point:	115.2°C at 760 mm Hg
Relative vapor density at 25°C:	3.190 (dry air = 1)
Relative density of saturated air at 25°C:	1.049 (dry air = 1)
Vapor pressure:	17.02 mm at 25°C
Solubility:	5.9% by weight in water at 20°C
At 25°C and 760 mm Hg:	
1 ppm vapor:	0.003781 mg/liter
1 mg/liter:	264.4 ppm
saturated air	22,390 ppm

III. Industrial Hygiene Practice

- A. **RECOGNITION:** Four humans detected the presence of 16 ppm of epichlorohydrin but only two could identify it. All four subjects recognized the odor at 64 ppm and one found this concentration to be irritating to the pharynx after two minutes.³ In another study, half of a group of unconditioned personnel detected 10 to 12 ppm, and all detected 25 ppm within five minutes. The threshold for eye and nose irritation was reported to be greater than 100 ppm.⁴ Epichlorohydrin is a primary raw material in the manufacture of epoxy resins and is also used in the production of other resinous materials.
- B. **EVALUATION OF EXPOSURE:**
 1. **Instrumentation:** A portable, battery-operated gas analyzer and a detector tube are available from at least one instrument supplier.
 2. **Chemical Analysis:** Analysis may be carried out by the method described by Daniel and Gage.¹ This sensitive colorimetric method for vapor of epichlorohydrin is based on oxidation with periodic acid followed by reaction of the formaldehyde produced with ammonia and acetylacetone to give a yellow color.
- C. **RECOMMENDED CONTROL PROCEDURES:** All persons handling epichlorohydrin should be instructed in its hazardous properties and in preventive measures. Completely closed systems are desirable plus general and local exhaust ventilation where necessary. Routine air analyses are essential. All possible effort should be made to avoid breathing the vapor and to prevent skin and eye contact with the liquid. Epichlorohydrin is known to per-

meate rubber; therefore, gloves and other rubber equipment should be evaluated carefully to determine their resistance to epichlorohydrin. Respiratory protection should be available for emergency use in spills or repairs.

IV. Specific Procedures

- A. **FIRST AID:** Remove person to uncontaminated atmosphere, remove soiled clothing and flush contaminated areas with plenty of water for 15 minutes. In case of an eye splash, irrigate 15 minutes with water or 0.9% sodium chloride solution and refer to a physician at once.
- B. **SPECIFIC MEDICAL PROCEDURES:** Individuals with pre-existing lung, liver, or kidney disease should not be placed in areas where there is risk of repeated or substantial exposure to epichlorohydrin. Regular medical evaluation of all exposed persons is indicated. Any exposed person with acute symptoms should be seen by a physician at once and placed at bed rest. The earliest symptoms of intoxication may be referable to the gastrointestinal tract (nausea, vomiting, abdominal discomfort, or pain in the region of the

liver). Labored breathing, coughing, and cyanosis may be evident. All further contact with the compound should be prevented and kidney functions followed closely. Oxygen and other supportive therapy for chemical pneumonitis should be instituted.^{4, 6}

V. References

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3. Pozzani, U. C., et al.: Unpublished data, Mellon Institute, Pittsburgh 13, Pennsylvania.
4. Shell Chemical Corporation: *Industrial Hygiene Bulletin* SC:57-86—*Epichlorohydrin*; Industrial Hygiene Department, 50 West 50th Street, New York 20, New York (1959).
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Ethylene Chlorohydrin

(2-Chloroethanol)

I. Hygienic Standards

- A. **RECOMMENDED MAXIMUM ATMOSPHERIC CONCENTRATION (8 hours):** Five parts of vapor per million parts of air, by volume (ppm).¹ Others have recommended 2 ppm.^{2, 6}
 1. *Basis for Recommendation:* Industrial observations and results of single animal inhalation exposures.¹⁰
- B. **SEVERITY OF HAZARDS:**
 1. *Health:* High for both acute and chronic exposure. Little margin of safety between early reversible symptoms and fatal intoxication. Absorption by any route, including the skin, may lead to severe illness or death. Contact with vapor causes irritation of mucous membranes, nausea, vomiting, vertigo, incoordination, numbness, and visual disturbance. With higher concentrations; headache, severe thirst, delirium, low blood pressure, collapse, shock,

and coma may result. Urine may contain albumin, casts, and red blood cells. Death results from pulmonary edema or from congestion and edema of brain.¹² The cause of the toxic effect is probably the result of oxidation of the alcohol to chloroacetic acid.¹³

- C. **SHORT INHALATION TOLERANCE:** 250 ppm (0.8 mg/liter) lethal to rats in four hours; 125 ppm (0.4 mg/liter) not lethal.⁸ 365 ppm (1.2 mg/liter) for two hours lethal to one of five mice.⁴
- D. **ATMOSPHERIC CONCENTRATION IMMEDIATELY HAZARDOUS TO LIFE:** Air saturated at room temperature lethal to rats in less than five minutes (ca. 6440 ppm or 4.9 mg/liter).⁸ A human fatality has been reported following exposure to 305 ppm (1 mg/liter) for 2¼ hours.⁴

II. Significant Properties

Colorless liquid of faint alcohol-etherlike odor
Chemical formula: $\text{ClCH}_2\text{CH}_2\text{OH}$

Molecular weight:	80.52
Boiling point:	128.7°C at 760 mm Hg
	60.0°C at 50 mm Hg
	32.0°C at 10 mm Hg
	-62.6°C
Freezing point:	-62.6°C
Vapor pressure:	6.8 mm Hg at 25°C
Specific gravity:	1.2045 20°/20°C
Relative vapor density:	2.77 (air = 1)
Relative density of saturated air at 25°C:	1.016 (air = 1)
Flash point:	140°F (open cup)
At 25°C and 760 mm Hg:	
1 ppm of vapor:	0.00329 mg/liter
1 mg/liter vapor:	304 ppm
saturated air:	8950 ppm
Solubility:	infinitely soluble in water, alcohol, benzene; less soluble in butyl acetate, castor oil and sulfonated oils

III. Industrial Hygiene Practice

- A. RECOGNITION: Warning properties are poor and inadequate to prevent injury. Its primary use is the introduction of the hydroxyethyl group ($-\text{CH}_2\text{CH}_2\text{OH}$) into other organic compounds.
- B. EVALUATION OF EXPOSURE:
 1. *Instrumentation*: No direct reading instruments available as yet, but a continuous conductivity analyser is suggested by Rowe.⁹
 2. *Chemical Method*: The concentration in air may be estimated by absorption of the vapor in potassium hydroxide solution, hydrolyzing it, and determining amount of chloride ion liberated.^{5, 7}
- C. RECOMMENDED CONTROL PROCEDURES: The high degree of hazard by inhalation makes it mandatory to keep the atmospheric concentration below 5 ppm. Routine air analyses are essential. Skin contact is particularly dangerous because the absence of immediate irritation prevents any warning when skin is wetted. (Quantitatively, the toxicity for guinea pigs is actually slightly greater by skin penetration than by the peroral route.) Rubber gloves do not provide adequate protection from undiluted or from aqueous solutions. Operations and processes must be planned to prevent contact with skin and vapor

by enclosure and/or ventilation.^{3, 11} Respiratory protection should be available for emergency use in repairs or spills.

IV. Specific Procedures

- A. FIRST AID: A physician should be called in all cases where overexposure is suspected. If swallowed, induce vomiting by forcing finger down throat or giving emetic. Remove clothing wetted by the chemical and flush contaminated skin with copious amounts of water. In case of eye splash, refer to physician at once after flushing eyes for 15 minutes with water or physiological saline. Administer artificial respiration and oxygen if necessary. Epinephrine is contraindicated because of the possibility of inducing ventricular fibrillation.¹²
- B. SPECIAL MEDICAL PROCEDURES: Persons with pre-existing lung, heart, liver, or kidney disease should not be placed in areas where there is any risk of exposure in excess of the 2 ppm threshold limit. Regular medical examinations of all exposed persons should be carried out.

V. References

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13. Williams, R. T.: *Detoxication Mechanisms*, John Wiley & Sons, New York (1959).

Methyl Chloride

(Chloromethane)

I. Hygienic Standards

- A. RECOMMENDED MAXIMUM ATMOSPHERIC CONCENTRATION (8 hours): 100 parts of vapor per million parts of air, by volume (ppm).

1. *Basis for Recommendation*: Principally animal studies.^{1, 6} Information is lacking on effects in humans exposed regularly to known concentrations.

- B. SEVERITY OF HAZARDS:^{2, 10, 11}

1. *Health*: High for acute and probably for chronic exposures. Deaths have occurred following single severe or repeated prolonged moderate exposures. While methyl chloride has relatively mild anesthetic properties, it produces a characteristic delayed central nervous system effect (as is also the case with methyl bromide). Symptoms consist of dizziness, vomiting, and stupor. Convulsions may be present and severe. Disturbances of vision may occur. Evidence of renal or hepatic damage and anemia may be found. The mortality may be as high as 35% and recovery prolonged.^{4, 6} The cause of the toxic effect is probably not conversion to methanol, as it appears to be largely excreted in expired air, urine, and bile.¹²

Repeated moderate or mild exposures may result in delayed (24-48 hours) onset of complaints of dizziness, headaches, mental confusion, slurred speech, diplopia, and somnolence. As in the case of severe poisoning, complete recovery may be prolonged. Skin burns may result from contact with the liquid form of methyl chloride.

2. *Fire*: Moderate. Flammable gas. Flammable limits in air are 8.25 to 18.7 volume per cent. Burns with a white flame.³

- C. SHORT EXPOSURE TOLERANCE: Exposures

to levels exceeding 500 ppm by volume in air may be dangerous.⁹

- D. ATMOSPHERIC CONCENTRATION IMMEDIATELY HAZARDOUS TO LIFE: Not known. Guinea pigs exposed to 95,000 ppm were unable to walk after two minutes and died 1-4 days later.⁸

II. Significant Properties

Colorless flammable gas at room temperatures and pressures with practically no odor.

Chemical formula: CH_3Cl

Molecular weight: 50.49

Boiling point: -24.2°C

Relative vapor density: 1.741 (air = 1)

At 25°C and 760 mm

Hg:

1 ppm of vapor 0.0021 mg/liter

1 mg/liter of vapor 484 ppm

Solubility: Methyl chloride gas/volume of water at 20°C and 760 mm Hg is 2.2 volumes. Soluble in most organic solvents.

III. Industrial Hygiene Practice

- A. RECOGNITION: Used as a refrigerant, expanding agent for plastic foams, dewaxing solvent, propellant solvent for insecticides, methylating agent and catalyst solvent in synthetic rubber production. Odor is faint, sweet, ethereal in high concentrations, but is practically odorless in concentrations of hygienic significance.

- B. EVALUATION OF EXPOSURES:

1. *Direct Reading Instruments*: Non-specific but satisfactory instruments include: electroconductivity analyzers, hydrogen flame ionization instruments, the Davis Halide Meter and halide leak detectors. (Limit of sensitivity of leak detectors is about 50 ppm, and above 200 ppm flame color is not a reliable index of methyl chloride concen-

The Committee wishes to acknowledge the assistance of Fred S. Venable in the preparation of this Hygienic Guide.

trations.) If specificity is necessary, the mass spectrometer, infrared spectrometer in combination with the gas chromatograph may be used. Electroconductivity and infrared methods are suitable for continuous monitoring of the atmosphere.

2. *Chemical Methods:* Methyl chloride may be determined by direct combustion in a quartz or Vicor tube⁵ or by burning an air sample with natural gas in a microburner.⁷ Products of combustion from either method are scrubbed with an aqueous basic reducing solution to trap the chloride ion which is then estimated by methods such as the micro-Volhard. Detector tubes by various manufacturers can probably be used, but each batch should be separately calibrated. None of these methods are specific for methyl chloride.

C. **RECOMMENDED CONTROL PROCEDURES:** All persons potentially exposed should be instructed in the hazard and in precautions. Maintain air concentrations below 100 ppm by enclosure of processes and, where necessary, by local exhaust and/or general ventilation. Affinity of methyl chloride for adsorbents is poor; therefore, chemical cartridge respirators or canister masks are not recommended for protection against unsafe levels. Supplied air respirators, approved by the U. S. Bureau of Mines, should be used for levels exceeding 500 ppm. Leakage of methyl chloride within buildings presents an insidious and serious hazard. Frequent monitoring or continuous sampling systems with alarm devices are recommended. Water containing dissolved methyl chloride should be handled in closed systems, or provided with local exhaust and/or general ventilation. Protection should be provided against contact with liquid methyl chloride.

V. Specific Procedures

- A. **FIRST AID:** Remove person to fresh air and call a physician. Keep the patient under medical observation for at least 48 hours. Oxygen and artificial respiration may be used in the event of respiratory difficulty.

B. **SPECIAL MEDICAL PROCEDURES:** The physician should consider carefully the placement of persons with a history of renal, blood or nervous system diseases. In the event of an acute exposure, the person should be followed medically for several days, with special attention to central nervous system signs and blood or renal changes. Hospitalization is desirable in the event of high level exposures or definite symptoms. All persons with repeated exposures should have routine medical examinations.

V. References

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2. Hansen, H., N. K. Weaver, and F. S. Venable: *AMA Arch. Ind. Hyg. and Occ. Med.* 8: 328 (1953).
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5. Jacobs, M. D.: *Analytical Chemistry of Industrial Poisons, Hazards and Solvents*. 2nd Edition, Interscience Publishers, Inc., New York (1949).
6. Johnstone, R. T., and S. E. Miller: *Occupational Diseases and Industrial Medicine*. W. B. Saunders Co., Philadelphia, Pa. (1960).
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12. Williams, R. T.: *Detoxication Mechanisms*. John Wiley & Sons, New York (1959).

Nitroethane

I. Hygienic Standards

A. RECOMMENDED MAXIMUM ATMOSPHERIC CONCENTRATION (8 hours): 100 parts of vapor per million parts of air, by volume (ppm).

1. *Basis for Recommendation:* Principally observations on animal inhalation exposures.^{1, 2, 3} Studies on humans exposed for long periods at these levels are lacking.

B. SEVERITY OF HAZARDS:

1. *Health:* Moderate for both acute and chronic exposures. Absorption is chiefly from inhalation. High concentrations produce respiratory tract and eye irritation, symptoms of narcosis and death in animals. All animals dying from single or repeated inhalation exposures or oral doses showed liver and kidney injury. Small groups of rabbits, guinea pigs, and one monkey survived daily 6-hour exposures to 500 ppm for a total of 140 hours. No skin injury or absorption was noted in animals. No blood changes or methemoglobin were noted.² Nitroethane apparently is metabolized to acetaldehyde and nitrite; some appears unchanged in unexpired air. Elimination is fairly complete in 30 hours.⁴ A mercapturic acid metabolite has been found.¹⁴ The most important effect of nitroethane is the development of organ damage, centered primarily in the liver from single or repeated doses. No injuries in man have been reported. Mild skin irritation in plant use resembles that of other solvents.⁶

2. *Fire and Explosion:* Moderate. Flash point (closed cup) 28°C (82°F),⁷ (open cup) 41°C (106°F). Lower explosive limit 4.0%. Nitroethane is quite stable to severe shock at ordinary temperatures. In tests made by the Bureau of Mines, nitroethane was detonated by a heavy charge of tetryl when it was confined in a heavy walled steel container and the temperature was raised to 100°C; it could not be detonated in a lighter walled container nor at ambient temperatures. Mixtures of undiluted

nitroethane with amines plus heavy metal oxides are very hazardous. Dry or highly concentrated alkalis form salts which decompose explosively if heated or subjected to shock in the dry state.^{6, 8, 9} Nitrolic acids are formed when primary nitroparaffins react with nitrous acid and salts of these acids are explosive when dry.¹⁰ Although nitroethane cannot be classified as a highly active chemical, caution is advised in trying new reactions or mixtures until the safety of the reaction has been established.⁶

C. SHORT EXPOSURE TOLERANCE: Animals survived single exposures at 2500 ppm for three hours. Strong eye and respiratory irritation was shown by animals at 500 ppm and above.²

D. ATMOSPHERIC CONCENTRATION IMMEDIATELY HAZARDOUS TO LIFE: Unknown: Animals have died when exposed at 30,000 ppm for 30 minutes, and at 5000 ppm for two hours.

II. Significant Properties

Nitroethane is an oily liquid with a relatively mild odor.

Chemical formula:	$\text{CH}_3\text{CH}_2\text{NO}_2$
Molecular weight:	75.07
Specific gravity:	1.045 (25°/4°C) (pure) 1.042 to 1.047 (25°/25°) (commercial)
Boiling point:	114.0°C
Freezing point:	-90°C
Vapor pressure (at 25°C):	20.6 mm
Solubility:	soluble in alkalis and many organic solvents, solubility in water 4.5% by volume at 20°C

At 25°C and 760 mm

Hg:	
1 ppm of vapor:	0.003 mg/liter
1 mg/liter of vapor:	326 ppm
Saturated air concentration:	27,100 ppm
Relative density of saturated air:	1.04 (dry air = 1.00)
Relative density of vapor:	2.58 (dry air = 1.00)

The Committee wishes to acknowledge the assistance of R. H. Dewey in the preparation of this Hygienic Guide.

III. Industrial Hygiene Practice

- A. **RECOGNITION:** Nitroethane is a solvent for many resins and gums and is used in organic synthesis. The vapors are noticeably irritating at concentrations above 100 ppm, but cannot be relied on for warning properties.
- B. **EVALUATION OF EXPOSURE:**
1. *Instrumentation:* None.
 2. *Chemical Method:* Collect in dilute sodium hydroxide and develop color with ferric chloride in acid solution,^{11, 13} or collect in methanol and develop color with p-diazobenzenesulfonic acid.¹²
- C. **RECOMMENDED CONTROL PROCEDURES:** Maintain atmospheric concentrations below 100 ppm by enclosure of the process or local exhaust ventilation. Supplied air or self-contained respirators should be used where high concentrations of nitroethane vapor may be encountered; nitroparaffins have a high heat of absorption on activated carbon and can start a fire in contact with Hopcalite catalyst, so respirators containing these materials should not be used.⁶ Activated carbon should not be used in a solvent recovery system for the same reason.

IV. Specific Procedures

- A. **FIRST AID:** Remove from exposure, remove contaminated clothing, cleanse skin with copious quantities of water; eye splashes should be irrigated for at least 15 minutes. In case of pulmonary symptoms, place at bed rest immediately and give oxygen. Obtain medical attention at once.
- B. **SPECIFIC MEDICAL PROCEDURES:** Persons exposed to concentrations approaching or exceeding the threshold limit value should have replacement and periodic medical

evaluation with special attention directed toward liver and kidney function and chronic respiratory disease.

V. References

1. American Conference of Governmental Industrial Hygienists: *Amer. Ind. Hyg. Assoc. J.* 22: 325 (1961).
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14. Williams, R. T.: *Detoxication Mechanisms.* J. Wiley & Sons, New York (1959).

Nitromethane

I. Hygienic Standards

- A. **RECOMMENDED MAXIMUM ATMOSPHERIC CONCENTRATION (8 hours):** 100 parts of vapor per million parts of air, by volume (ppm).
1. *Basis for Recommendation:* Principally results of repeated animal inhalation

studies.^{1, 2, 3, 4} Data on the effects in humans exposed repeatedly at these levels over long periods is lacking.

B. SEVERITY OF HAZARDS:

1. *Health:* Moderate for both acute and chronic exposures. Absorption is chiefly from inhalation. High concentrations produce evidence of mild respiratory tract irritation, then narcosis followed by death. The most important effect

The Committee wishes to acknowledge the assistance of R. H. Dewey in the preparation of this Hygienic Guide.

of single or repeated vapor exposures in several species of experimental animals is systemic toxicity with damage centering chiefly in the liver and to some extent to the kidney. Liver and kidney damage and mild respiratory tract irritation develop before narcotic symptoms occur.^{2, 3, 4} No blood changes or methemoglobin formation were noted.² There is little conversion to nitrite *in vivo*.¹⁶

Small groups of rabbits, guinea pigs, and a monkey survived daily 6-hour exposures to a total exposure of 140 hours.² The liquid is not irritating to animal skin. There is no evidence of skin absorption.

There have been no reported injuries in man. Mild skin irritation observed in plant use has been attributed to the solvent action on the skin.⁶

2. **Fire and Explosion:** Moderate. Flash point (closed cup) 35°C (95°F),⁷ open cup 44.4°C (112°F).⁶ Lower explosive limit 7.3%

Nitromethane is quite stable to shock in ordinary handling but can be detonated when confined in a heavy walled container and subjected to a shock from a large dynamite cap or a charge of tetryl.^{8, 9} Tests indicate that the shock must be sudden enough and the confinement great enough to cause adiabatic compression of gas or gas bubbles in the container which will initiate thermal decomposition. The shock sensitivity will be increased by the addition of aniline, ethylene diamine, and similar alkaline materials and can be decreased by dilution with other solvents.⁶

Mixtures of undiluted nitromethane with amines plus heavy metal oxides are very hazardous because of the formation of fulminates. Dry or highly concentrated alkalis form salts which decompose explosively if heated or subjected to shock in the dry state.^{6, 9, 10} Nitrolic acids are formed when nitromethane reacts with nitrous acid, and salts of these acids are explosive when dry.¹¹ Caution is advised when trying out new mixtures or reactions with nitromethane until the safety of the reaction has been established.⁶

- C. **SHORT EXPOSURE TOLERANCE:** Animals survived single exposures at 10,000 ppm for one hour. Severe eye and respiratory

irritation was noted in animals at 500 ppm and above.²

- D. **ATMOSPHERIC CONCENTRATION IMMEDIATELY HAZARDOUS TO LIFE:** Unknown. Animals have survived a single exposure at 30,000 ppm for 15 minutes but have died when exposed at the same concentration for 30 minutes.²

II. Significant Properties

Oily liquid with a moderately strong, somewhat disagreeable odor.

Chemical formula:	CH ₃ NO ₂
Molecular weight:	61.04
Specific gravity:	1.132 (25°/4°C) (pure) 1.124 (25°/4°C) (commercial)
Boiling point:	101.2°C
Freezing point:	-29°C
Vapor pressure (at 25°C)	35.5 mm Hg
Solubility:	soluble in alkali and many organic solvents, solubility in water 9.5%, by volume at 20°C

At 25°C and 760 mm

Hg:	
1 ppm of vapor:	0.0025 mg/liter
1 mg/liter of vapor:	401 ppm
Saturated air concentration:	46,600 ppm
Relative density of saturated air:	1.05
Relative density of vapor:	2.11

III. Industrial Hygiene Practice

- A. **RECOGNITION:** Nitromethane is a solvent for many resins and cellulose esters and is used in the coating industry. It is used as an intermediate in the synthesis of many organic compounds. The odor is very strong above 100 ppm and irritating at 200 ppm,² but cannot be relied on for warning properties.

- B. **EVALUATION OF EXPOSURE:**

1. **Instrumentation:** None.
2. **Chemical Methods:** Collect in alkali and develop color with vanillin;² collect in methanol and develop color with p-di-azobenzenesulfonic acid;¹² collect in dilute sulfuric acid and determine polarographically;¹³ collect in buffered-phosphate solution and couple with

diazotized ethylaminobenzoate and pyridine to form a red dye.^{14, 15}

- C. **RECOMMENDED CONTROL PROCEDURES:** Maintain atmospheric concentrations below 100 ppm by enclosure of the process or local exhaust ventilation. Supplied air or self-contained respirators should be used where high concentrations of nitromethane vapor may be encountered. Nitromethane has a high heat of absorption on activated carbon and oxidizes vigorously in contact with Hopcalite catalyst, so respirators containing these materials should not be used.⁶ Activated carbon should not be used in a solvent recovery system for the same reason.

Equipment should be designed to avoid sudden and adiabatic compression,⁶ and detonation traps should be installed on both sides of a pump handling nitromethane.⁹

IV. Specific Procedures

- A. **FIRST AID:** Remove from exposure, remove contaminated clothing, and cleanse skin with copious amounts of water. Eyes should be irrigated for at least 15 minutes. In cases of pulmonary distress, place at bed rest immediately, and give oxygen. Obtain medical attention at once.
- B. **SPECIFIC MEDICAL PROCEDURES:** Individuals exposed to concentrations approaching the threshold limit value should be offered preplacement and periodic medical evaluations with special attention directed toward liver and kidney function.

V. References

1. Threshold Limits List, American Conference of Governmental Industrial Hygienists: *Amer. Ind. Hyg. Assoc. J.* 22: 325 (1961).
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Propanol

(Propyl Alcohol)

The two isomeric forms, n-propanol (propanol-1, n-propyl alcohol) and isopropanol (propanol-2, isopropyl alcohol) are similar in physical and in most physiological properties. Isopropanol is in more common use.

I. Hygienic Standards

- A. **RECOMMENDED MAXIMUM ATMOSPHERIC CONCENTRATION (8 hours):** 400 parts of vapor per million parts of air by volume

(ppm) for isopropanol.¹ A value has not been established for n-propanol.

1. **Basis for Recommendation:** Human sensory data and analogy with ethyl alcohol.^{2, 3}
- B. **SEVERITY OF HAZARD:**
 1. **Health:** Low for both isomers, for both acute and chronic exposure. The main effect from inhalation or ingestion is narcosis.^{4, 5, 6} There is very little risk

for humans from the inhalation of the vapors or either of these isomers from industrial processes,⁶ because high concentrations are required to induce a narcotic effect. In contrast to ethanol, isopropyl alcohol does not cause a primary stimulation when ingested, but only a depression.⁵ Salivation, retching, and vomiting are said to be more frequent than with ethanol.⁶

Part of the isopropanol absorbed by the body is oxidized to acetone and excreted as such,^{4, 5} whereas n-propanol is oxidized completely in the body.⁵ Animals exposed repeatedly to anesthetic concentrations (about 10,000 ppm) of vapors of these alcohols recovered with no serious aftereffects.¹⁴

2. *Fire and Explosion:* Moderate. Limits of flammability:⁷ n-propanol 2.1 to 13.5%, isopropanol 2.0 to 12%. Flash points (closed cup): n-propanol 15°C (59°F) isopropanol 11.7°C (53°F). Saturated air concentrations of these alcohols are within the explosive range.

- C. *SHORT EXPOSURE TOLERANCE:* Very high concentrations can be tolerated with some discomfort and irritation of the eyes. Rats exposed to saturated air mixtures of isopropanol showed only slightly toxic symptoms after 15 to 30 minutes.⁸ Inhalation experiments with mice indicate that 4000 ppm (10 mg/liter) can be tolerated for an hour without any signs of incoordination.⁸

- D. *ATMOSPHERIC CONCENTRATION IMMEDIATELY HAZARDOUS TO LIFE:* Unknown, but well above the saturated air concentration at ordinary temperatures. Animals exposed to saturated air concentrations have all survived.^{5, 8} Mice exposed to concentrations of 24,000 ppm (60 mg/liter) of isopropanol for 100 minutes showed no aftereffects.⁵ Since the saturated air concentrations are explosive, the explosion hazard would be greater than the health hazard at very high concentrations.

II. Significant Properties

Both isomers are colorless, flammable liquids with moderately strong alcoholic odors.

	n-propanol	isopropanol
Chemical formula:	$\text{CH}_3\text{CH}_2\text{CH}_2\text{OH}$	$\text{CH}_3\text{CHOHCH}_3$

	n-propanol	isopropanol
Molecular weight:	60.1	60.1
Specific gravity (20°/4°C)	0.804	0.785
Boiling point; 760 mm Hg:	97.8°C	82.5°C
Vapor pressure, at 25°C:	21 mm Hg	43.5 mm Hg
Relative vapor density (dry air = 1)	2.07	2.07
Relative density of saturated air at 25°C (dry air = 1)	1.03	1.06
Air saturated at 25°C and 760 mm of Hg contains:	2.76% (by vol)	5.72% (by vol)
Solubility:	both isomers are infinitely soluble in water, ethanol, and ether	
At 25°C and 760 mm Hg:		
1 ppm of vapor:	2.46 mg/m ³	
1 mg/liter:	407 ppm	

III. Industrial Hygiene Practice

- A. *RECOGNITION:* n-Propanol is used as a laboratory solvent and in some organic syntheses; its use in industry is limited. Isopropanol is used mainly for the manufacture of acetone, but is also used in cosmetics and toiletries, for some cleaning and dehydrating operations, and has a limited use in the paint and lacquer industry. The odor of isopropanol is difficult to recognize in the presence of other solvents or odorant materials and is readily masked.

B. EVALUATION OF EXPOSURE:

1. *Instrumentation:* Direct reading detector tubes available on the market will give a reasonable estimate of propanol concentrations.
2. *Chemical Methods:* Collection in water in an all glass device with subsequent determination by dichromate oxidation.^{9, 10, 11} Isopropanol has been determined in urine by oxidation to acetone and determination of the acetone.¹² The red complex formed between hydroxyl groups and ceric ammonium

nitrate¹³ might also be used. A combination of methods may be needed for none of these are specific for the propanol isomers.

- C. **RECOMMENDED CONTROL PROCEDURES:** Atmospheric concentrations should be kept below 400 ppm to prevent discomfort, although individuals can tolerate up to 800 ppm without serious discomfort.³ If the solvent is heated or used in enclosed areas, local exhaust ventilation is required.

Eliminate potential sources of ignition where the propanols are handled, because n-propanol and isopropanol, and also their water solutions, will flash at room temperature.

IV. Specific Procedures

- A. **FIRST AID:** If symptoms are noted following severe exposure, remove to fresh air. If splashed in the eyes, wash eyes with copious amounts of water. Spills of anhydrous alcohols on skin or clothing should be diluted promptly under a safety shower.
- B. **BIOCHEMICAL ASSAY:** n-Propanol is apparently completely oxidized in the body,⁵ although a portion is probably excreted through exhaled air. Isopropanol is partially eliminated unchanged through the exhaled air and in the urine, and can be determined by appropriate chemical methods.^{5, 12} Isopropanol is partly metabolized to acetone which is excreted in the exhaled air and in the urine,^{4, 5} and this can be determined.
- C. **SPECIFIC MEDICAL PROCEDURES:** No special preplacement or other examinations appear necessary because of the innocuous character of these alcohols.

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Cumene

(Isopropylbenzene, Isopropylbenzol, Cumol, 2-Phenylpropane)

I. Hygienic Standards

- A. **RECOMMENDED MAXIMUM ATMOSPHERIC CONCENTRATION** (8 hours): No official

The Committee wishes to acknowledge the assistance of Dr. M. L. Keplinger, Dr. J. P. Frawley, and Dr. Horace Gerarde in the preparation of this Hygienic Guide.

value. A suggested tentative value is 100 parts of vapor per million parts of air, by volume (ppm).

1. **Basis for Recommendation:** Observation of experimental animals and comparison of effects to those of toluene,

xylene, and ethylbenzene, which have established threshold limits.^{1, 2, 3}

B. SEVERITY OF HAZARD:

1. **Health:** Moderate. Absorption occurs chiefly by inhalation of vapors or mist. High concentrations irritate the respiratory tract and produce narcosis. Chronic exposure of rats at about 500 ppm caused congestion of lungs, liver, and kidneys, but no bone marrow damage.¹ Prolonged or repeated contact of liquid with the skin may cause dermatitis. There are no reports of systemic toxic effects in humans. The odor and irritating characteristics of the vapor at low levels tends to limit exposures.
2. **Fire:** Moderately flammable, definite hazard of fire and explosion. Flash point 38.8°C (102°F) (TCC).

- C. **SHORT EXPOSURE TOLERANCE:** Occasional short exposures to concentrations of 400 ppm probably would not be harmful.⁴ LC₅₀ of cumene vapors to mice is 2040 ppm (single 7-hour exposure)⁵ and to rats is 8000 ppm (single 4-hour exposure).⁶ On a volume basis, comparison of acute narcotic effects of cumene vapors in mice show that cumene is about two times as active as vapors of toluene and about five times as active as benzene.

- D. **ATMOSPHERIC CONCENTRATION IMMEDIATELY HAZARDOUS TO LIFE:** Exposure to substantially saturated vapor at room temperature killed none of five rats in two hours, two of six rats in four hours, and six of six rats in eight hours.⁷

II. Significant Properties

A clear, colorless liquid with a sharp, penetrating aromatic odor detectable at very low levels. Contact with tongue causes painful burning sensation with profuse salivation.

Chemical formula:	C ₉ H ₈ CH(CH ₃) ₂
Molecular weight:	120.19
Specific gravity:	0.85748 (25°/4°)
Boiling point:	152.4°C (306.3°F)
Vapor pressure:	4.5 mm Hg at 25°C
Per cent in saturated air, 760 mm:	0.58 at 25°C
Relative vapor density:	4.2 (air = 1 at 25°C)
Relative density of air saturated at 25°C:	1.019
Solubility:	very limited in water, soluble in ethanol and water

At 25°C and 760 mm

Hg:

1 ppm of vapor:	0.00492 mg/liter
1 mg/liter of vapor:	203.5 ppm

III. Industrial Hygiene Practice

- A. **RECOGNITION:** Cumene has a characteristic odor. It is used in aviation gasoline as a high octane blending component, in thinners for paints and enamels, as an intermediate in the manufacture of phenol and acetone and as an intermediate in synthesis.

B. EVALUATION OF EXPOSURE:

1. **Instrumentation:** Direct determination by the use of aromatic hydrocarbon detector tubes sold by various manufacturers. Collection by scrubbing through spectroscopically pure ethanol in an all-glass device, followed by ultraviolet spectroscopy.
2. **Chemical Method:** Collection by absorption on silica gel followed by nitration similar to that for the determination of toluene,⁸ or treatment with sulfuric acid-formaldehyde mixture (if interfering aromatic hydrocarbons absent). For determination in blood, an ultraviolet spectrophotometric method may be used.¹

- C. **RECOMMENDED CONTROL PROCEDURES:** Maintain workroom atmosphere below 100 ppm by means of closed system and/or adequate ventilation. Prevent gross skin contact through use of protective clothing or gloves impervious to aromatic hydrocarbons. Use supplied air mask in cleaning of tanks or for cleaning up accidental spillage of large amounts in a relatively confined area.

IV. Specific Procedures

- A. **FIRST AID:** Cleanse eyes (15-minute wash with running water) and skin if direct contact with liquid. Remove patient from contaminated area. Administer artificial respiration immediately if breathing stops. Oxygen may be administered; Epinephrine is contraindicated. Vapors may cause irritation of eyes, mucous membranes and skin. Discontinuation of exposure is usually sufficient.
- B. **SPECIAL MEDICAL PROCEDURE (Including Preplacement):** Persons with chronic skin disease or definite abnormalities of the blood, liver, kidney, or lungs should not

be placed at work where there may be a possibility of substantial or repeated exposure to cumene. Regular medical examinations of exposed personnel are desirable.

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ACCIDENTAL DEATHS IN AGRICULTURE

THE DIVISION OF VITAL STATISTICS of the Kansas State Board of Health has recently published an 18 page booklet presenting a study of accidental deaths reported among Kansas farm workers during 1960. The purpose of the publication is to provide facts that show the extent of the farm work accident problem as depicted in terms of deaths involved. The facts presented show that many of the fatal accidents could have been avoided.

The total number of deaths in Kansas attributed to occupational agricultural accidents last year is 55. This is the highest since 1955 when an equal number died and is 11.1% higher than the average for the last ten years. Although farm fatalities during the 1951-60 period declined 34.5% from the previous ten-year period, deaths from the operation of farm tractors climbed by 15.8%. The report shows conclusively that careless use of the tractor presents by far the greatest hazard to the farmer today. Tractors accounted for 34 of the 55 deaths, or 61.8%. Overturning was the cause of almost two-thirds of the tractor fatalities.

The report breaks the death statistics down into almost every conceivable system of classification, showing the deaths by specific causes, sex, age, time of day, season of the year, and by county. The report also provides the basis for farm safety education and is available upon request to the Division of Vital Statistics, Kansas State Board of Health, State Office Building, Topeka, Kansas.

News of Local Sections

Pacific Northwest Section

The sixth annual meeting of the Pacific Northwest Section was held September 22 and 23 at Idaho Falls, Idaho. The climax to the varied program offered to 53 meeting registrants was a tour of the Experimental Breeder Reactor No. 2, operated by Argonne Laboratory, which was approaching criticality at the time.

The annual Joseph Dalla Valle Award was presented by the Section to Mr. Frank Adley.

New officers for 1962 are: President, John R. Horan; President-Elect, Willson C. Applegate; Secretary-Treasurer, Bruce J. Held; Directors, Darrel Douglas and Donovan E. Wisehart.

An Industrial Ventilation Conference is planned for June, 1962, at Seattle, with the University of Washington as co-sponsor.

Western New York Section

Mr. Lee Schreibeis of the Bethlehem Steel Company was the speaker at the Section's Fall Meeting held the evening of September 29 at the Sheraton Hotel in Buffalo. His subject was "Current Thoughts on Air Pollution." Also on the program was a movie entitled "Hospital Sepsis" which portrayed the ways bacteria might be airborne throughout a hospital.

An afternoon meeting and program of several papers is planned for December 9.

Northeastern Michigan Section

A dinner meeting was held November 8 in cooperation with the Greater Saginaw Safety Council. Dr. David E. Harmer of the Dow Chemical Company's Nuclear and Basic Research Laboratory spoke on "Living with Radiological Defense."

The Section plans additional meetings next February and April or May, the latter meeting to be held at Ann Arbor with the other Michigan sections.

Ohio Valley Section

The Ohio Valley Section has a new slate of officers as the result of the annual election held in June 1961. Robert H. Starkey (National Lead Company) assumed the Presidency, having been President-Elect. Howard E. Ayer (U.S. Public Health Service, Division of Occupational Health) was named President-Elect, and J. J. Schuene-

man (U.S. Public Health Service, Division of Air Pollution) was elected Secretary-Treasurer. Lawrence J. Schafer (Kettering Laboratory) was selected as a new director.

The Section has affiliated with the Technical and Scientific Societies Council of Cincinnati. The purpose of the Council is to coordinate activities of technical societies in relation to those matters of a professional, educational, or public nature which are beyond the scope of activity of individual societies or which can be performed by cooperative action. In addition to professional benefits, association with the Council provides for periodic use of excellent meeting facilities.

More than forty Section members attended the first meeting of the 1961-1962 season. The group toured a brewery, observing the sanitation, safety, occupational health, and production aspects of the plant. Suitable testing of the products of the establishment was accomplished by the usual sensory methods. A dinner meeting was held in October at the Technical and Scientific Societies Building. New developments in national air quality monitoring were described by Mr. E. C. Tabor of the Division of Air Pollution of the U.S. Public Health Service.

Tennessee Valley Section

The annual Fall Meeting of the Tennessee Valley Section was held at the Atomic Energy Building, Arnold Engineering Development Center, Tullahoma, on October 6, 1961. The full day's program extending from registration at 8:30 A.M. through a dinner at 6:30 P.M. included the presentation of five technical papers and a tour of various laboratories and activities of the research center. Papers presented were "Health Aspects of the Handling and Storage of Fluorine" by Henry P. Heubusch, "Protection in Industrial Radiography" by Peter J. Valaer, "Preliminary Studies in the Chemical Composition of the Respirable Fraction of Air-borne Coal Dust" by W. O. Bionconi and E. D. Myers, "Ventilation Improvements to Relieve Heat Stress" by Raymond H. Wagner, and "Control of Health Hazards from Tunneling Operations" by E. D. Myers.

Officers for the coming year are D. P. Roberts, President; R. B. L. Bleming, Past-President; James F. Morehead, President-elect; Robert H. Wolle, Secretary-Treasurer; and E. P. Marconi, Director.

The Section plans to hold their Spring Meeting in Oak Ridge concurrent with the meeting of the Industrial Hygiene Fellowship Committee for the AEC. With this group of prominent national members as guests, a dinner meeting with a most interesting program is anticipated.

Philadelphia Section

The regular meeting of the Philadelphia Section was held at the Engineers Club on September 12, 1961. The social hour was followed by an excellent dinner and a short business meeting. The speaker for the evening was Norman G. White, Ph.D., Shell Chemical Corporation. Dr. White presented a very interesting and comprehensive discussion of the epoxy resins and the

problems which are encountered with the use of these materials in industry today.

Utah Section

This Section sponsored and provided a booth at the Utah State Fair. This booth was set up to enlighten the general public on the industrial hygiene program, its functions and responsibilities. Approximately 30,000 people visited the booth over a seven-day period and the booth was shown and discussed for 15 minutes on a local television broadcast. The booth showed lung sections, sampling equipment, radiation detecting equipment, models of dust collecting equipment, and other environmental control equipment.

MICROSCOPIC ANALYSIS

A TRAINING COURSE in *Microscopic Analysis of Atmospheric Particulates* will be conducted January 22 through February 2, 1962, at the Robert A. Taft Sanitary Engineering Center in Cincinnati. It is presented by the Public Health Service through its Division of Air Pollution. The course emphasizes the application of chemical microscopy techniques to qualitative and quantitative identification of specific particulates. Laboratory sessions, which include more than half of the course time, give practice in the determination of the physical properties of small solid and liquid particulates, in methods of evaluation by size distribution, and in microscopic spot test procedures. Discussions cover phase and electron microscopy. Instruction is handled exclusively by Dr. Walter C. McCrone, director of the McCrone Research Institute in Chicago. Dr. McCrone is nationally known in the field of chemical microscopy and is the author of many publications. The course annually attracts chemists, engineers, and other professional people responsible for or interested in identification of airborne particulates. No registration or tuition fee is required.

Applications for the course or requests for information may be addressed to the Chief, Training Program, Taft Sanitary Engineering Center, 4676 Columbia Parkway, Cincinnati 26, Ohio, or to a PHS Regional Health Director.

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